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CONTENTS

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Topics of the Month

Chemical stockpiling

THE extent to which raw material stockpiling is being carried out by the chemical industry is very clearly shown by the January trade accounts published last month. Almost every item is being imported in quantities considerably above those of the comparable month last year. Total value of imports was no less than £4,719,514, compared with £2,609,934 for January 1950 and £2,175,503 for January 1949. Even allowing for the increase in prices during the past twelve months, it is obvious that imports are rising to feed the stockpiles.

Among the most notable increases are the following, the figures for January 1950 being given in brackets: boric acid 12,830 cwt. (10,550), borax 38,901 cwt. (28,440), calcium superphosphate 46,275 tons (11,045), glycol ethers and glycol ether-esters 507,376 lb. (182,038), iodine 88,118 lb. (16,000), potassium chloride 799,119 cwt. (573,416), potassium sulphate 50,362 cwt. (27,760), turpentine 35,447 cwt. (15,446), petroleum waxes 61,833 cwt. (39,421) and mercury 83,612 lb. (28,864).

The most notable decrease, of course, is sulphur—29,223 tons (32,285). This smaller quantity, it might be added, cost £369,245, against £307,197 for the much bigger imports a year previously.

If stockpiling on this scale continues, the chemical industry will be in reasonably good shape to meet an emergency, with the exception of sulphur.

Radiochemical school

TO many scientists the thought of using radioactive chemicals in research creates a picture of vast and elaborate laboratories equipped with intricate and expensive equipment. This picture is undoubtedly derived from descriptions and illustrations of the lavishly equipped laboratories of the atomic energy research establishments at Harwell and Amersham. True enough, for some kinds of tracer research such arrangements are necessary. It is equally true, however, that experiments with radioactive tracers can be performed with simple equipment costing no more than a few score pounds and consisting principally of a Geiger-Müller counter and scaler. Only recently we saw a demonstration of the use of tracers to follow the track of an insecticide through the sap system of a plant. The 'props' consisted of the plant in a flower pot and a small G-M counter. The demonstration was given during lunch in a West End hotel!

It is unlikely, however, that words will be sufficient to convince the sceptics. It is necessary to familiarise as many scientists as possible with the methods before they become generally accepted in industry. A good step in this direction has been taken by the Ministry of Supply, who have organised an atomic energy school to teach workers in industrial and medical laboratories how to use radioactive materials. The first course began on April 2 at the A.E.R.E., Harwell. It will last six weeks. Subsequent courses will last four weeks. Twelve students will be accepted on each course which will include 12 to 18 lectures. The syllabus will be varied slightly to meet the requirements of the majority. The courses are designed to give students sufficient knowledge of the principles underlying work with radio-isotopes to qualify them to start work in their own special fields or to undertake research calling for specialised techniques.

The second and third courses will begin on May 15 and June 18. Further details can be obtained from Dr. J. E. Johnston, Isotope Division, A.E.R.E., Harwell, Berks.

New South African oil refinery

SINCE the war there has been a progressive reversal of the former policy of the great oil companies of refining petroleum on or comparatively near the oilfields. The most striking example of this new trend is here in Britain where, in spite of insignificant indigenous reserves of oil, a huge refinery capacity is being built up based upon supplies of crude from overseas.

South Africa is the latest country to follow this new trend. A few weeks ago the Government there joined with the Vacuum Oil Co. of South Africa Ltd. in announcing plans for a refinery near Durban. This plant, which will be the first in South Africa to process a natural crude oil, will have a capacity of some 10,000 barrels/day and will cost about $12,500,000. This money is being provided by the American parent company, Standard-Vacuum Oil.

The plant will be essentially a fuels refinery, turning out products ranging from motor spirit to heavy bunker fuel. It is now in the drawing board stage. Process designs and construction plans are being developed and it is expected that the contract will be awarded within a few months and that ground-breaking operations will start early next year. The refinery should be ready to go on stream late in 1953 or early in 1954.

It is likely that the refinery will incorporate several new operating principles. It will probably consist of a single integrated unit in which will be combined the functions of distillation naphtha reforming and fluid catalytic cracking. The usual facilities for chemical treatment, storage tanks for working stocks, shops, laboratories, administrative offices, etc., will be installed.

The refinery will be built and operated, utilising South African sub-contractors, materials and supplies as much as possible, by a new company, Standard-Vacuum Refining Co. of South Africa Ltd. It will occupy a 214-acre site at Wentworth, the industrial suburb of Durban. Selection of Durban as the site was largely due to the deep-water facilities for tankers which will carry the refinery's crude. Vacuum already has a marine terminal at Durban, which will be enlarged and eventually connected with the plant by a $4\frac{1}{2}$-mile pipeline. An obvious possible source of the crude is the Persian Gulf area, although this has not yet been settled.

Coal through pipes

IF an American experiment succeeds, coal will become as easily transportable as oil and gas; that is, it will be possible to move it through pipes. Pittsburgh Consolidation Coal Co., said to be the largest soft-coal producer in the world, has conceived the idea of pumping coal as a slurry through a 12-in. pipe. A demonstration system, with a pipeline 17,000 ft. long, is to be built in eastern Ohio at a cost of some $550,000.

The system will work as follows: After mining, coal will be washed and crushed to fine size and then mixed with water to form a slurry. The slurry will be fed into the pipeline and moved through under pressure by specially designed pumps. Equipment at the end of the line will remove the coal and then dry it.

The demonstration system will be operated continuously and will be able to handle several thousand tons of coal daily. The pipeline will be built near Cadiz, Ohio, at the company's Georgetown strip-mining facilities. The company hopes eventually to extend its plan to general commercial use.

The Indian salt industry

PROGRESS towards the sorely-needed expansion of the Indian salt industry has reached the stage of discussion of a five-year development programme by representatives of the industry with Government officials. The discussions took place at a recent meeting of the Planning Commission.

Of a total number of salt-making units in the country with an annual production capacity of about 2,600,000 tons, six units with a capacity of 740,000 tons are owned and operated by the Government, and 27% of the land area used for salt manufacture is under Government operation. Capital invested in the salt industry, according to the Salt Experts Committee's report, is estimated at 60,000,000 rupees. Maximum production was attained during 1950, with an estimated output of 2,627,000 tons. The programme suggested by the Committee aimed at a consumption target of 3,000,000 tons by 1955, against a potential production of 4,000,000 tons.

Steps to improve production and quality of salt which were discussed seem to be based upon the report of the committee of salt experts mentioned above, which was noted in these columns in January (p. 14). They include consolidation of small fields and realignment of units, elimination of manual labour wherever possible through mechanised handling, and establishment of model Government factories at different centres. The committee has also suggested the establishment of a statutory corporation to operate Government-owned salt works.

Safe handling of ammonium nitrate

IN April 1947 more than 500 people were killed when two freighters loaded with ammonium nitrate caught fire and exploded in the harbour of Texas City. The Monsanto Chemical Co.'s $20,000,000 styrene plant and a large part of the city were destroyed, the damage being increased by the setting off of a train of explosions involving benzene and toluene in the styrene plant. In July of the same year, a third vessel loaded with ammonium nitrate exploded off the coast of Brest, France. These disasters resulted in several separate investigations into the reasons why a comparatively safe chemical suddenly became dynamite in every sense.

In the U.K. the Home Office examined the effect of fires on stored ammonium nitrate. Tests were made on the island of Dune, near Heligoland, but negative results were obtained, no explosion occurring despite the severity of the fires and large amounts of ammonium nitrate involved.

In France transport regulations were laid down stipulating that ammonium nitrate could be loaded in bulk in trucks or metal boats, provided it was not in contact with wood, or in wooden trucks, provided the sides and bottom were completely coated with impermeable, fireproof lining or with sodium silicate. Precautions were also to be taken to prevent the risk of heating and contact with inflammable materials and it was forbidden to leave vessels loaded with ammonium nitrate inside the Paris area and within other designated zones.

In the U.S., the Texas City catastrophe resulted in the formulation of extremely restrictive emergency regulations. With a view to having these regulations remodelled in the light of present knowledge, the Manufacturing Chemists' Association, Inc., has produced an excellent booklet dealing with the packaging, handling, transport, storage and use of this important fertiliser.

This Texas City incident is discussed at some length and, it is stated, ' A complete review of the experience in handling

millions of tons of the material has demonstrated that the safe handling of this product requires the same kind of precautions as those which have been well established for the safe handling of other oxidising materials. The fires and explosions aboard ship at Texas City resulted from improper handling and ineffective fire-fighting.

'As in all activities where possible hazards exist, the most important thing is to determine what causes the danger and then prevent the development of such a condition. In the case of fertiliser-grade ammonium nitrate, this is readily accomplished by following the practices recommended in the booklet. Fertiliser-grade ammonium nitrate, like many other commodities of everyday use in the city and on the farm, is safe if properly handled.'

A number of fires involving stored fertiliser-grade ammonium nitrate are on record, the booklet reports, but 'investigation of all commercial and farm storage fires does not reveal a single case in which an explosion of ammonium nitrate occurred.'

The following recommendations for safe handling and storage of the material are made: (1) Keep away from heat and open flame; (2) Do not store with combustible materials; (3) In case of fire, flood with water; (4) Burn empty bags promptly in the open; (5) Place broken or torn bags and contents in overslip bags; (6) Sweep up and dispose of all spilled material immediately; (7) Do not rebag loose material.

In addition to handling and storage practices, the booklet discusses the chemical and physical properties of ammonium nitrate and recommended transportation methods, warehousing and how to fight an ammonium nitrate fire. Also included is a partial bibliography of pertinent literature.

Increasing Europe's iron ore production

EXPERTS on iron ore production and consumption have been examining Europe's requirements for iron ores. At a meeting held in Geneva by the Iron Ore Working Group of the United Nations Economic Commission for Europe, the principal ore-producing and exporting countries indicated that 3,400,000 more tons of ore would be available for import in 1953 than previous figures had shown, but this would still leave a deficit. The experts asked countries to supply new data for a more detailed picture which would show quality-by-quality estimates of ore requirements and availabilities for 1953.

Because of the relationship of ore requirements to supplies of coke and scrap, the other two basic materials for iron and steel production, the experts hope before their next meeting to obtain information on prospective metallurgical coke supplies for 1953. Countries are also being asked to provide information on the rate of scrap consumption anticipated for 1953 in their blast furnaces.

According to present plans, Europe expects to produce 15% more pig iron in 1953 than it did in 1950. While many ore-producing countries are planning for greater output of ores, the development of ore production is a matter of years and not months. That is why the governments, in the framework of E.C.E., are concentrating on ore production in 1953 and after. They hope, by gauging demand prospects for that date, to give indications to producers which would help them in their planning. It is, of course, also important for countries planning further expansions of steel plants to be sure of availabilities of raw materials, including a knowledge of prospective sources and qualities.

In its effort to ensure adequate supplies of iron ore, the Working Group has now undertaken the first Europe-wide study of prospective supplies for the major categories of the ores (i.e. lean and rich iron ores, high- and low-phosphorus ores, etc.).

Countries participating in this group are Austria, Belgium, Czechoslovakia, France, Italy, Netherlands, Sweden, Switzerland, the U.K. and the U.S.A.

German pharmaceuticals

THE value of West German pharmaceutical exports in 1950 totalled 90,599,000 marks, compared with only 26,208,000 marks in 1949. Before the war about 38% of the world's pharmaceutical exports came from Germany; the value of her exports in 1936 was 108,113,000 Reichsmarks. Last year's great progress was made in spite of many difficulties, chiefly the loss of trade marks and patent rights abroad. The problem of legal protection of German trade marks and patent rights is still unsettled; for the time being it can only be tackled through bilateral agreements. The industry expects that most of the patents will not be restored, but as most of them have expired or are now out of date there is little concern on this score. The restoration of trade marks is far more important. The industry has tried to reach a general settlement on this, but prospects do not seem very favourable. In any case, manufacturers doubt whether the old trade marks will ever regain their value after being used for many years by foreign competitors. The introduction of new trade marks, however, would be difficult.

Domestic market conditions were largely normal in the past year after a sales crisis in 1949 resulted in over-stocking. The value of turnover in 1950 was estimated at 420,000,000 to 430,000,000 marks, compared with about 400,000,000 marks in 1949. Raw material supplies were adequate until a few months ago, as about 95% of them are domestically produced. But now manufacturers are worried about the growing coal shortage, which might seriously hamper production of both raw materials and finished products. Supplies of ampoules, bottles and other packing materials are also short.

The pharmaceutical industry of the former Reich was largely concentrated in the west. Nearly all factories in that part of Germany which is now the Soviet zone have been nationalised. Many former owners of expropriated plants in the east established new factories in West Germany, but they now have to fight competition from their old plants, which continue to produce under the old name and trade marks and try to smuggle their goods into West Germany. The East German industry, however, is not regarded as a serious competitor in foreign markets, for its production is negligible.

The West German industry is mainly situated in the Rhein-Main area (Frankfurt, Weisbaden, Darmstadt), in North Rhine-Westphalia (Leverkusen) and at Hamburg. The leading firms include Farbwerke-Hoechst, of Frankfurt, E. Merck, Darmstadt, C. H. Boehringer, Ingelheim, and Chemische Werke Albert, Wiesbaden.

Farbwerke Hoechst, formerly part of the I.G. Farben combine, was among the first pharmaceutical factories to resume production after the war. Up till then, pharmaceuticals made in I.G. Farben plants were exclusively marketed by the Bayer-Leverkusen firm under the trade mark of that firm, so the Farbwerke Hoechst had to set up a new sales organisation. Apart from some other new products turned out by the Farbwerke after the war, large-scale production of penicillin—about 400,000, mega units per month—began in the summer of 1950 (see INTERNATIONAL

CHEMICAL ENGINEERING, November 1950, p. 492). The Farbenfabriken Bayer at Leverkusen, also once a part of I.G. Farben, is working again at full capacity. Its main products are the sulphonamides, among them the famous *Prontosil*, and penicillin. Other products include *Contaben* for treating tuberculosis and some anti-histamines.

P. Beiersdorf A.G. of Hamburg produces mainly plasters. Production in 1950 exceeded that of 1939. The chemical factory Promonta G.m.b.H., also at Hamburg, produces cosmetics, as well as pharmaceuticals. Since April the firm has manufactured an ACTH product named *Cortiphyson*. Schuelke and Mayr A.G. at Hamburg, chiefly engaged in disinfectants, once more produce *Lysol* and *Sagrotau*. Last year they also developed *Lysolin*, claimed to be a non-poisonous and odourless disinfectant.

Imports of pharmaceuticals, negligible before the war, amounted in value to 32,813,000 marks in 1950, against 17,378,000 marks in 1949. They consisted chiefly of penicillin, insulin and natural drugs.

Robot ear controls tube mill feed

THE usual method of pulverising coal for modern draught-fed furnaces and boilers is to feed raw coal into a tube mill in which it is reduced to powder by steel balls cascading within the rotating tube or drum. The efficiency of this process is almost wholly dependent upon the correct amount of raw coal being maintained within the mill, in other words, upon accurate control of the coal feed.

Hitherto, this control has been performed by a skilled operator who relied upon his highly developed sense of hearing. If he was tired or had a cold in the head his sense of hearing and, in turn, the process suffered.

Automatic control was the obvious answer to the risk of human failing, but conventional methods, including mechanical ones, have failed to give satisfaction. Standard Telephones & Cables Ltd. were recently asked to investigate the problem, and they have solved it by imitating the human ear with an acoustic and electrical device.

In their investigations, the company discovered that to be effective any means of automatic feed control must be based upon the actual, and not estimated, amount of raw coal within the mill. Thus their acoustic device is designed to determine the actual contents of the mill from the noise level of the cascading coal and balls in contact with each other and with the walls of the drum.

As with the human ear, this calls for extremely fine distinctions in noise level taken at a reference point in the immediate vicinity of the drum.

The basic apparatus consists of a microphone, adequately protected against dust and dirt, which is placed immediately beneath the drum. This is connected by cable to the main controller unit, which is set up at any desired distance from the tube mill.

The output stage of the controller unit comprises a series of pads for lining-up purposes, an amplifier and a high-pass filter, the function of which is to segregate the significant high-frequency noises emanating mostly from inside the drum, from the low-frequency noises associated chiefly with the external gearing and drive of the mill itself.

It is a tribute to the engineers concerned that they have evolved a device able to make this delicate distinction between sound levels which has proved so effective that it is already scheduled for installation in a number of fuel processing plants.

Although the acoustic mill feed controller has been engineered for the special job of coal pulverising, it seems that it could be applied to tube mills processing other materials such as minerals and chemicals.

We understand, however, that this would be possible only if the mill is operated continuously; it is ineffective with batch processing, and this may limit its application in the chemical industry.

World Refrigeration Congress

REFRIGERATION is a process which has always been of fundamental importance in chemical engineering and lately its significance in this connection has increased with the wider application of low temperatures to modern chemical processes. The many scientific and industrial aspects of what has been called 'the art of producing cold' will be discussed at the 8th International Congress of Refrigeration which is to be held in London from August 29 to September 11. This congress, the first to be held since 1936, will reflect the great progress made in this branch of engineering since the time, 50 years ago, when the great German refrigeration pioneer, Dr. Carl von Linde, called Great Britain 'the cradle of refrigeration.' The many refrigeration engineers and technicians from all over the world who are expected to attend the congress will have the opportunity of seeing the application of refrigeration to all manner of industrial plants, from steel production and petroleum refining to nylon manufacture and a host of other chemical and industrial processes.

During the first week of the congress in London, daily meetings will be held to receive and discuss the papers and reports contributed from many countries. Excursions to refrigeration installations and works in and around London and official visits to the Festival of Britain will also be made. The technical board of the International Institute of Refrigeration, under whose auspices the congress is held, will also meet. During the second week, industrial tours to different provincial areas have been arranged.

Other events of interest to refrigeration engineers taking place in Britain at this time will be the Heat Transfer Conference of the Institution of Mechanical Engineers, open to congress delegates, and a Low-Temperature Physics Conference of UNO.

Further details of the congress and membership forms may be obtained from the secretary, 8th International Congress of Refrigeration, Dalmeny House, Monument Street, London, E.C.3.

Water-gas from coal slurry

AS pointed out by Drs. Hall and Jolley of the Fuel Research Station, in their paper on the industrial possibilities of the fluidised-solids technique (INTERNATIONAL CHEMICAL ENGINEERING, April 1950, pp. 159-166), the use of the fluid-bed principle could provide a way of producing water-gas from low-quality, small-size fuel now largely regarded as a waste product. According to a recent statement from the Department of Scientific and Industrial Research, the Fuel Research Station is now operating an experimental fluid-bed plant which is converting coal-washery slurry into water-gas. At present, water-gas produced from the usual starting material, high-grade coke, costs 7d. per therm. It is estimated that if the new process is successful, this cost could be reduced by 50%, at the same time releasing 1,250,000 tons of coke annually for industrial and domestic use. Besides water-gas, the process could yield fuel oil and other useful hydrocarbons.

A New Approach to Coal Tar as a Source of Chemicals

By A. Fowler-Williams, M.Sc., A.R.I.C., A.M.Inst.F.

(Central Research Establishment, National Coal Board)

Although there is much loose talk about coal being 'a vast storehouse of organic chemicals,' the author believes that too little is being done too slowly to exploit fully the chemical potentialities of coal tar. The position is greatly different in the petroleum industry and great projects for producing chemicals from crude oil are under way in Britain and elsewhere. In spite of the lead taken by the petroleum industry, the author argues that the coal industry has not lost its opportunity to provide a wide range of chemicals. The technological and economic basis for this view he presents in the following article.

RAPID advances in the scale of the demand both at home and abroad, for the products mainly of the plastics, paint, detergents and synthetic fibre industries, have necessitated the production of increasing quantities of organic chemicals to serve as raw materials, intermediates and solvents. A large part of the requirements, particularly such solvents as the aliphatic alcohols, glycols, ketones, etc., are derivatives of olefinic hydrocarbons which can be produced by cracking the larger molecules of compounds present in natural petroleum.

At the same time, cracking can be made to occur with a varying degree of aromatisation within the residual oil, so that compounds such as benzene, xylene, naphthalene, anthracene and other higher-boiling polynuclear hydrocarbons known to exist in coal tar are now being made available from petroleum materials.

Furthermore, since the production of certain chemicals from the natural sources, petroleum and coal tar, is often costly and, in any case, insufficient to meet market demands, there has been an increasing tendency to synthesise the required compounds from simpler and cheaper intermediates. Thus, the greater part of the demand by the plastics and paint industries for styrene monomer is being met by the dehydrogenation of ethyl benzene synthesised from ethylene and benzene (both arising from petroleum or coal gas). Again, the production of phenol from coal tar is quite insufficient to meet the demands of the plastics (phenol-formaldehyde resins) and synthetic fibre (nylon) industries, and the process of synthesis from benzene is now well established on a commercial scale.

It is clear that the coal tar industry, as constituted at present, may come to play a relatively insignificant part in the production of chemicals or indeed of any materials competitive with petroleum products. The purpose of the present paper is to point out the opportunities for a fuller exploitation of the chemical potential of coal tar, emphasis being placed on compounds known to be present, but not
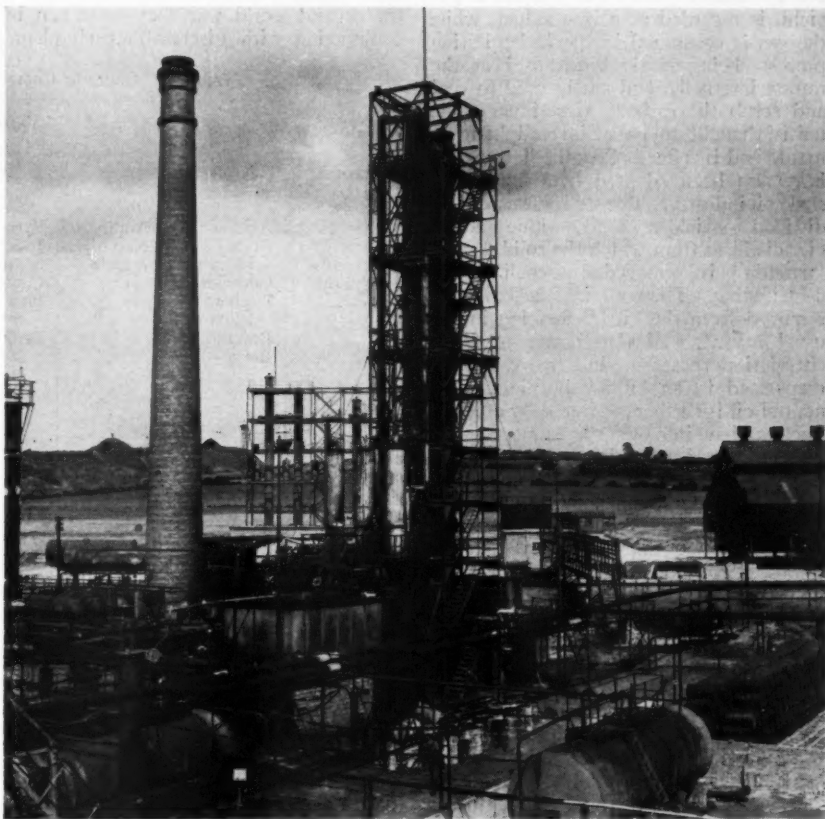
Crude oil distillation section of the plant operated at Bolsover by Coalite & Chemical Products Ltd., with whose permission the photograph appears. The unit is described in column 2, p. 161.

extracted in quantity on a commercial scale.

Coal and coal tar as a source of chemicals

The tar supplies in Great Britain result mainly from two major high-temperature coal carbonisation industries, of which the principal products are foundry or domestic coke and gas for towns' supply respectively. The physical, and more particularly the chemical, nature of such tars varies with the nature of the parent coal and the conditions under which this is carbonised. Thus, although the qualitative nature of 'high-temperature tars' obtained from coke ovens and gasworks may be similar, the quantitative spread of chemicals may be markedly different in the two cases. Further, the chemical composition of high-temperature tar is widely different from that of 'low-temperature tar' such as is

obtained by Coalite & Chemical Products Ltd. in the manufacture of the domestic smokeless fuel (coke) known as *Coalite*.

The German low-temperature carbonisation industry

Low-temperature carbonisation has been developed into a tremendous industry in Germany, where the raw material, mainly brown coal, is carbonised at temperatures between 550 and 750°C. by the Lurgi-Spülgas and Krupp-Lurgi processes, the latter being suitable for bituminous coals also. The *brown coal* industry dates back about 100 years and developed mainly alongside plants for extracting montan wax prior to carbonisation at Riebeck, Amsdorf and Wansleben. The singular feature of the brown coal industry is that the products derived from the tar form the economic backbone of the process. The coke, although obtained in a much larger yield, is regarded as a by-product, while the gas is consumed in the carbonisation process, being partly burnt to heat the retorts internally, and partly used to cool and scrub the carbonised residue. The tar is thought to be mainly aliphatic in nature and is refined along the lines which have also been adopted by the Scottish shale oil industry. The tar is destructively distilled to yield a coked residue and an oily distillate from which the solid wax or ' paraffin ' is removed by cooling and centrifuging. The tar acids and bases are removed from the oil by washing in the usual manner and the former used for chemical purposes or, in some cases, incorporated in an oil for burning. The neutral oil is further separated by efficient fractionation into petrol, kerosene and diesel oil fractions, which are up-graded if necessary, by solvent extraction (liquid sulphur dioxide) to remove the unsaturated and aromatic materials. The coke was used for the production of carbon electrodes, but the main product of the industry was wax-steam candles.

During the 1939-45 war, tar from brown coal became the principal raw material for the production of petrol by hydrogenation, and in 1944 amounted to about one-seventh of the total liquid-fuel production. The semi-coke was used for the production of water-gas in specially designed producers and removal of the carbon monoxide fur-

nished hydrogen for the hydrogenation plants.

Processes of low-temperature carbonisation based on *bituminous coal* developed much more slowly in Germany and were never really economic. However, during the second world war they were run in conjunction with Fischer-Tropsch plants

for the primary purpose of making oil from coal. The yield of tar (of which a typical analysis is shown in Table 1) was about 6% on the dry coal. About 95% of the tar was sold as boiler fuel for industrial purposes and, during the war, it was sold as fuel oil to the German Navy, which required a minimum S.G. of 1.03.

TABLE 1. ANALYSIS OF TAR OBTAINED BY LOW-TEMPERATURE CARBONISATION IN GERMANY

Specific gravity at 20°C.	1.06
Viscosity at 20°C. ..	40 Engler
„ „ 50°C. ..	5 „
Flash point °C. ..	96
Tar acid content % ..	25—30
Initial boiling point °C.	98
Final boiling point °C.	360
Distilled to 170°C. %	1.5
„ „ 300°C. %	55
„ „ 360°C. %	66
Calorific value	16,200 B.Th.U./lb.
Free carbon (insoluble in benzene) % ..	0.7
Pitch content % ..	35

TABLE 2. PRODUCTS FROM COAL-OIL

Product	Main uses	Crude oil %
Phenol	Plastics, nylon, etc.	1.00
Ortho cresol	Antiseptics, etc.	0.25
Meta/para cresol	Plastics, antiseptics, etc.	2.25
Xylenols	Insecticides, plasticisers, disinfectants.	4.00
High-boiling tar acids ..	Froth flotation, insecticides, etc.	14.50
Diesel oil	Road transport.	22.00
Washed light oil ..	Solvent for rubber, paints, etc.	3.00
Cresylic pitch	Briquetting.	4.00
Paraffin wax	Candles, di-electrics, waterproofing of paper, etc.	1.50
Fuel oils	———	
Fluxing oils	———	
Residues	———	47.5
Loss	———	

TABLE 3. ORGANIC COMPOUNDS PRESENT IN COAL TAR.

Oxygen compounds		Nitrogenous compounds					Sulphur compounds
Phenolic	Neutral	Amino				Nitrile	
		Primary	Secondary	Tertiary			
Phenol Cresols Xylenols Naphthols etc.	Acetone Aceto-phenone Coumarone Dipheny-lene oxide Brasan Benzo-xanthene etc.	Aniline Naphthyl-amine etc.	Pyrrole Indole Skatole Carbazole Phenan-thridone Benz-carbazole etc.	Pyridine Picolines Lutidines Quinoline Isoquino-line Acridine Phenan-thridine Benz-acridine etc.		Aceto-nitrile Benzo-nitrile Naphtho-nitrile etc.	Carbon disulphide Me and Et mercaptans Dimethyl sulphide Thiophene Thio-naphthene Dipheny-lene sulphide etc.

| Hydrocarbons						
Saturated		Unsaturated				
	Paraf-finic	Cyclic	Olefinic		Aromatic	Hydro aromatic	Aromatics with un-saturated side chains
			Mono	Di			
Ali-pha-tic	Pentane to nona-decane, etc.						
Nu-clear		Cyclo-hexane etc.	Cyclo-hexene etc.	Cyclo-pentadiene Cyclo-hexadiene Dicyclo-pentadiene etc.	Benzene Toluene Xylenes Naphthalene Methyl naphthalene Acenaphthene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benzfluorenes Chrysene Triphenylene Naphthacene Benzpyrenes Perylene Picene Crackene Benzerythrene Truxene, etc.	Tetralin Dihydro-anthracene Tetrahydro-fluoranthene Hydro-chrysene Perhydro-picene etc.	Styrene Indene Methyl indene Dimethyl indene
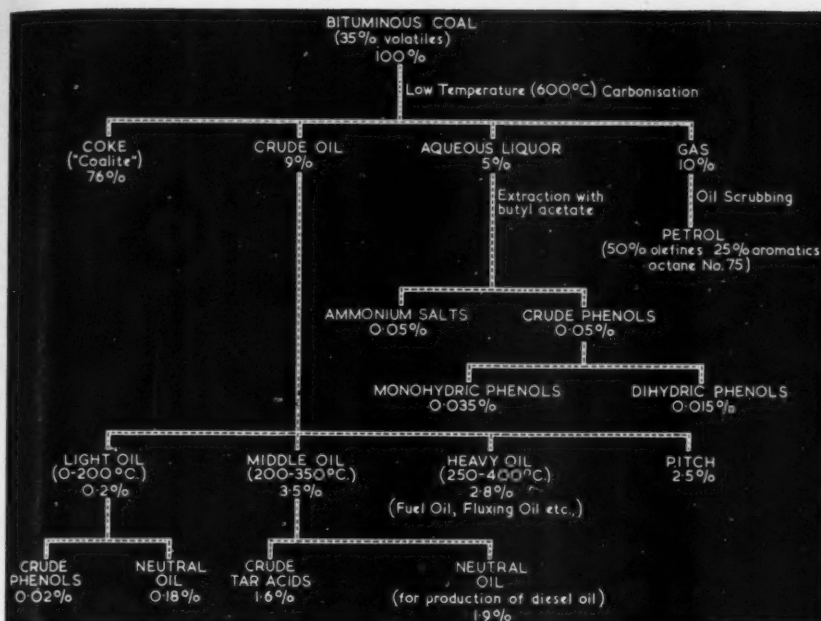
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Fig. I. Yields of chemicals from bituminous coal by the low-temperature carbonisation process.

The Krupp-Lurgi process of low-temperature carbonisation is applicable to British coals, particularly those of the coking variety. In Germany, the coke was used in closed domestic stoves and in central-heating plants or bakeries. However, on account of its high reactivity, it was found to be particularly suitable for making synthesis gas and thence petrol and diesel oils by the Fischer-Tropsch process. This process has never been developed seriously in the U.K., but rapid strides are being made in the U.S.A. towards a commercial process for making 80-octane petrol from natural gas. According to the exact conditions of temperature, pressure, nature of catalyst, etc., governing the synthesis, the yield of petrol, high-boiling oils, solid waxes and oxygenated bodies (alcohols, ketones, fatty acids, etc.) can be varied at will. In areas where natural gas is not available, the prime requirement for the economic working of the Fischer-Tropsch process is cheap coal for the raising of synthesis gas, so that it is doubtful whether this means of making aliphatic chemicals from coal will ever be adopted in the U.K. However, it is interesting that German industrialists are applying to the Allied Control Commission for permission to recommission the Fischer-Tropsch plants for the production of waxes and oxygenated aliphatic chemicals and hydrogen for synthetic ammonia.

Low-temperature carbonisation in England

It is gratifying that, despite the developments described previously, the Germans are far behind the stage that has been reached in this country in the production of chemicals from bituminous coal by the low-temperature carbonisation process.

This is mainly due to the research into the constitution of the liquid product that has been carried out since the first world war by Coalite & Chemical Products Ltd. The main product is a reactive coke of high volatility, but a substantial business has also been built up on the sale of petrol, diesel oil, tar acids and pitch. The crude oil distillation section of the firm's Bolsover refinery consists of two tube stills of conventional design arranged with fractionating columns 82 ft. high and containing 32 bubble decks at 2-ft. spacing. The crude oil is continuously distilled and divided up into light (–200°C.), middle (200 to 350°C.) and heavy (250 to 400°C.) oil and pitch, respectively. The light and middle oil fractions are treated with aqueous caustic soda solutions and the extracts distilled separately. The neutral oils are also redistilled so as to yield various useful oils, including one suitable for blending with standard gas oil of high cetane number so as to yield oil for diesel engines used in road transport vehicles. Approximate yields of products are shown in Fig. 1.

The late Col. Bristow (*J. Inst. Fuel*, 1947, **20**, pp. 109-127) outlined a modified scheme of refining, whereby the crude oil would be vacuum-distilled and the total distillate separated into tar acids and neutral oil, respectively. Further vacuum fractionation of the former should then yield the full range of tar acids. In this way the yields of products listed in Table 2 could be expected from the original coal-oil.

In this connection it is interesting to note that high-temperature tar (ca. 5% by weight of the coal carbonised) yields only 2 to 3% of tar acids, 0.5% of light oil, no paraffin wax and 50 to 60% of pitch.

High-temperature carbonisation

The quantitative changes which accompany coal carbonisation processes have been studied in detail by various workers, and considerable information has been accumulated concerning the physical characteristics of the by-products, coke, tar and gas. However, if one can judge from published literature, few serious attempts have been made to determine the redistribution and re-grouping of carbon which occurs within the major chemical-producing by-product, tar. The complexities of the entangled parent coal substances are unfolded in the various combinations, mainly between carbon and hydrogen and oxygen and, to a much lesser content, between carbon, hydrogen, nitrogen and sulphur also, which constitute some 200 single chemical components of coal tar. Exactly how the proportioning of these compounds is governed by the nature of the coal and the conditions of its carbonisation (temperature and retort design) is really only just beginning to be understood.

Some of the various types of compounds known to exist in coal tar are shown in Table 3.

However, although there has been much loose talk about tar being a 'vast storehouse of organic chemicals,' a distinction must be made between the compounds which have been used or can be extracted for use on a commercial scale and the very large number of 'curiosities' which are present in extremely small amounts and which have been isolated by various investigators during work on the constitution of tars.

Such chemicals as benzene, toluene, xylene, naphthalene, anthracene, phenol and the cresols, pyridine and its homologues, etc., are readily obtained by the refining of coal tar and command a wide and ready market. Smaller amounts of less well-known chemicals, including the polycyclic hydrocarbons, carbazole, diphenylene oxide, etc., also exist in tar and can be removed by efficient fractionation. However, since the demand for these 'special chemicals' appears to be problematical, few tar distillers bother to remove them. Thus, less than 10% of tar is used for strictly chemical purposes, while the remainder is sold as such relatively low-priced crudes as fuel oils, gas-scrubbing oils, wood preservatives, surface-dressing materials, bonding agents, etc.

It is being said that the coal tar industry has lost to the petroleum industry the opportunity of making a wide range of chemicals and that the lead could have been secured if only methods of efficient fractionation had been applied to coal tars in past years. The problem is one of supply and demand for, naturally, the tar distiller is not willing to go to the expense of isolating a particular chemical before the manner in which it is to be used is known. It may well be, however, that in the face of the changing situation, the chemical potential of tar will have to be exploited to the full

TABLE 4. CONSTITUENTS OF LOW-PRICED COAL TAR CRUDES

Primary coal tar fraction	Chemical	Theoretical boiling point at atmospheric pressure	Approx. boiling range of the appropriate fraction of coal tar	Theoretical m.pt. of pure compound	% in typical coke oven tars	Annual potential based on a total coke oven tar production (U.K.) of 1,000,000 tons
			At 100 mm. mercury			
Naphthalene oil	Naphthalene crystals	218°C.	137—158°C.	80.2°C.	8—9	ca. 85,000 t.
	Beta-methyl naphthalene	241		34.4		
	Alpha-methyl naphthalene	245	158—189	–30.8	4—5	ca. 45,000
	Diphenyl	255				
	Dimethyl naphthalenes	261—8				
Creosote →	Acenaphthene (crystals)	277	189—199	95.4	0.5—1.0	ca. 7,500
	Diphenylene oxide (crystals)	287	201—204.5	82.8	0.3—0.5	ca. 4,000
	Fluorene (crystals)	297	210—220	114.0	0.6—1.0	ca. 8,000
			At 50 mm. Hg.			
	Phenanthrene	340		100.0	2.0—2.5	ca. 22,500
Anthracene oil or pitch	Anthracene	342	222—249°C.	217.0	0.5—1.0	ca. 7,500
	Carbazole	355		245.0	ca. 1.0	ca. 10,000
	Fluoranthene	382	249—264	109.0	0.05—0.1	ca. 750
	Pyrene	393	266—278	151.0	ca. 0.1	1,000
	Benzfluorenes	398—401	278—310	189 (1.2) 208 (2.3)	ca. 0.05	500
	Chrysene	448	310	252	ca. 0.1	1,000

and the rarer chemicals put on the market at a price low enough to stimulate interest as to their possible industrial application.

With increasing demands for fine chemicals, the ' curiosities ' in coal tar have been explored and are being processed to the limit, e.g. the tar bases such as pyridine, *beta*-picoline, *ortho*-picoline and 2.6 lutidine, quinoline, isoquinoline, cyclopentadiene, styrene, indene-coumarone (usually as a resin mixture), pseudocumene, dicyclopentadiene, diphenyl, etc.

The fractions of coal tar which offer themselves for further investigation are the high-boiling, low-priced crudes such as naphthalene oil, creosote (benzene absorbing oil, wash oil, etc.) and anthracene oil. The predominant constituents of these fractions are given in Table 4.

Production technique

Table 4 shows that there is a sufficient separation between the boiling points at reduced pressure of about a dozen chemicals, to enable production by the technique of vacuum fractionation to be undertaken. In fact, the operating efficiency of the fractionating column can be so varied, by controlling the reflux ratio, as to enable the pure chemicals to be isolated directly by solvent extraction or one recrystallisation of appropriate distillates.

The major factor that influences the characteristics of a tar produced in different types of carbonising equipment is temperature, variations in the physical and chemical nature of the coal substance playing a relatively indifferent role. Low-temperature tar is thin and oily, containing a large proportion of primary tar, phenols, naphthenes and unsaturated compounds. The effect on the tar of increasing the carbonising temperature is to increase progressively its pitch content and also the content of aromatic chemicals up to an optimum concentration. The tar acid content decreases markedly, giving birth

to more benzene and naphthalene, while the paraffin and naphthenic contents decrease to raise the proportion of higher aromatics. Naphthalene and anthracene salts form between 700 and 1,000°C. and decompose between 1,000 and 1,100°C. and, in general, the overall aromatic content of a tar is greatest in that from a coal carbonised at 900°C. approximately. Few concerns in Great Britain rigidly adopt the standard method for observing carbonising temperatures, but a survey carried out by the British Coke Research Association showed that, in 1945, 34.5% of the total number of coke ovens were using a flue temperature of 1,250°C. or higher. The potential of highly aromatised tar in the country should therefore be considerable. At the very high temperatures, some of the chemicals undergo cracking or demethylation processes. The methyl naphthalenes certainly suffer some condensation and mutual elimination of methyl groups takes place to increase the yields of acenaphthene, fluorene and the higher aromatics found in pitch.

With regard to the production of suitably aromatised high-temperature tar on a commercial scale, three main types of retorts have been used. Vertical retorts

TABLE 5
PRODUCTS OF TWO TYPICAL ENGLISH TARS

	Weight of dry gas works tar %	Weight of dry coke oven tar %
Naphtha . .	10.6	2.72
Naphthalene crystals . .	4.9	8.4
Methyl and polymethyl naphthalenes . .	15.0	5.2
Acenaphthene crystals . .	0.27	0.56
Diphenylene oxide crystals	0.25	0.38
Fluorene crystals . .	0.37	0.70
Acenaphthene-diphenylene oxide-fluorene oils	3.45	1.67
Anthracene oil plus pitch	65.2	80.37

consisting of intermittently or continuously operated varieties are generally recognised to represent one type of high-temperature carbonising equipment. The combined tar which emerges from the continuously operated type of vertical retort contains tars which have been subjected largely to low-temperature conditions, to some extent to mid-temperature conditions and only to a small extent to high-temperature conditions. Its characteristics are intermediate between those of low- and high-temperature tars. On the other hand, coke ovens and horizontal retorts yield tars which are predominantly high-temperature in character, and anyone interested in the production of coal tar aromatic chemicals would therefore be advised to limit his choice to these two types. Table 5 compares two typical English tars carbonised at the same temperature (but arising from different coals), one being produced in a gas works (horizontal retorts) and the other in a coke oven plant. The comparison was made by batchwise vacuum fractionation of each tar under identical conditions.

Gas works tar, in containing a higher proportion of ' oily ' intermediates, is not so highly ' cracked ' or ' aromatised ' as coke oven tar, and this is further substantiated by the higher yield of pitch from the latter. In this respect tars arising from so-called petroleum aromatisation processes are similar to gas works tars in containing a relatively small proportion of solid aromatics.

The figures quoted in Table 4 for the possible annual production of chemicals from coke oven tar are conservative estimates, based on the author's own pilot-plant analyses of typical samples using methods of high-efficiency vacuum fractionation. It is certain that present-day practice of distillation in pot stills does not, for instance, extract the maximum amount of naphthalene. Naphthalene boils at 218°C. and is usually separated in the

200 to 230°C. fraction of coal tar. If proper fractionation methods were employed, one would not expect naphthalene to extend into the creosote fraction, but the author has found the crystals to account for about one-fifth of a coke oven creosote produced in a continuous-pipe still fractionating assembly. Thus, in this particular plant, the naphthalene output was capable of being raised by at least 2%. The figures in Table 4 show that the U.K. coke oven supplies alone are capable of yielding annually about 80,000 tons of naphthalene, a chemical for which the demand by the paints, plastics and dyestuffs industries exceeds the supply. However, only about 40,000 tons is at present produced annually from all available sources of coal tar. Again a considerable market exists for anthracene and about 3,000 tons are produced and sold every year, but an output of 7,000 tons could be reached without undue expansion in present-day refining practice.

Tar producers may scorn yields of chemicals of the order of 0.1%, but outputs of thousands of tons may be reached. It is true that no market exists at present for most of the products mentioned, but there can be little doubt that, if they were available to an expanding chemical industry in tonnage quantities, at a reasonable price and in a high degree of purity, research would take up the challenge and an increasing scale of consumption would follow.

The German high-temperature tar industry

Much valuable information on this subject was obtained by Allied scientific investigators working in Germany immediately after the second world war, their findings being published in the form of reports sponsored by the British, American and French Governments. Most of these reports have been summarised by A. L. Deadman and G. H. Fuidge in the B.I.O.S. Surveys Report No. 25 ('The German Coal Tar and Benzole Industries During the Period 1939-45'), from which, together with the report on his own investigation (B.I.O.S. Final Report No. 1783, Item No. 30, 'German Practice in the Production and Utilisation of High-Boiling Coal Tar Chemicals'), the present author now freely quotes.

The reports make it clear that the German tar industry has carried the commercial isolation of chemicals to greater lengths than the British tar industry, particularly in the years immediately preceding 1939. Large quantities of what we know in Britain as the usual coal tar chemicals (naphthalene, phenols, pyridine, etc.) were produced during the war, but smaller amounts of less well-known chemicals were isolated by methods of efficient fractionation. These chemicals commanded a ready market and included the bases pyridine, alpha-picoline, lutidines and collidines, lepidine, quinoline, isoquinoline, acridine, indole, methyl indoles and skatole; tar

acids such as phenol, *ortho* cresol, *meta-para* cresol mixture and the xylenols; and such 'oddities' as diphenyl, pseudocumene, dicyclopentadiene, thionaphthene and indene-coumarone mixture (sold as a resin for paints and varnishes). These more unusual chemicals represented only a small proportion of the tar, so that their isolation was commercially possible only at works where very large quantities of tar were being processed. However, the principle of centralised refining, coupled with the predominantly coke oven nature of the tar, gave the German tar chemical industry a distinct advantage.

The most important tar-distillation syndicates were Rutgerswerke A.G., Gesellschaft für Teerverwertung m.b.H. (G.F.T.), Chemische Werke Oberschlesien, G.m.b.H. and Vereinigte Stahlwerke. G.F.T. was the largest, with a capacity of 500,000 tons p.a. and processing at Duisberg-Meiderich, Rauxel and Alsdorf, 60% of the tar produced in the Ruhr. Comprehensive tar chemical production, especially of the rarer chemicals, was confined to G.F.T. and Rutgerswerke.

Possible outlets for coal tar chemicals

The tonnage outlets exist in the paints, plastics, dyestuffs and detergents industries. Research has already shown that *acenaphthene* can be converted into acenaphthylene, a valuable source of high softening point plastics, as well as into 1.8 naphthalene dicarboxylic acid, which simulates the valuable phthalic acid. *Pyrene* is known to be a useful source of green and light blue dyestuffs and of naphthalene tetra carboxylic acid, required by the plastics industry. In general, hydrogenated polynuclear aromatics such as *octahydrophenanthrene* or *tetra-hydrofluoranthene* have a potential value as plasticisers, especially in the preparation of dielectric plastic mixes, while the vinyl derivatives of the aromatics, *e.g. vinyl fluorene*, etc., might be used as intermediates for high softening polymers.

Alpha methyl naphthalene, freed from accompanying paraffins, has been used as a reference diesel fuel and also showed promise as an intermediate for naphthylacetic acid orchard sprays. On a lesser

scale, the *beta isomer*, in the form of the 1-4 quinone, was and is used today as vitamin K substitute and as a plant growth stimulant. The Germans used *diphenylene oxide* as an intermediate for the textile dyestuff Naphthol A.S. Brown and, by treatment with sodium, converted it into *ortho* hydroxy diphenyl, which was used in substantial quantities in the textile industry as a germicide. *Carbazole* was used for the manufacture of hydron blue (a valuable sulphur dye said to be the modern competitor of indigo), for polyvinyl carbazole ('Luvican') plastics and as tetra nitro carbazole or dinitrodichloro carbazole, as as base of *Nirosan* insecticides used against the vine moth. The methyl naphthalenes, acenaphthene oil, fluorene oil, etc., are useful high-boiling solvents, fluxing or heat-transfer media.

In 1943, I.G. Farben purchased from Rutgerswerke alone 263 tonnes of acenaphthene, 144 tonnes of diphenylene oxide, 127 tonnes of carbazole, 120 tonnes of pyrene and 8 tonnes of chrysene.

Coal tar v. petroleum

There can be little doubt that markets for existing coal tar crude products will be seriously affected by the outputs from oil refining and cracking plants soon to be brought into operation in the U.K. The coal tar fuel oil market which has persisted since the end of the second world war will undoubtedly be encroached upon by the petroleum oils with their superior calorific value, while bitumen will compete with pitch-creosote mixtures for road tars. There is room for more than one source of benzene, naphtha, naphthalene, etc., but in the face of a contracting market for creosote and other high-boiling coal tar oils, surely a case can be made out for investigating the possibility of producing the aforementioned chemicals from these fractions? In this the coal tar industry will have a clear advantage, for although the one known petroleum-aromatisation process produces high-boiling hydrocarbons ranging from naphthalene to chrysene, their proportion in the petroleum tar is much less than in coke oven tar (see Table 6).

It is true that the solid aromatics account

TABLE 6. COMPARISON OF YIELDS OF CHEMICALS FROM PETROLEUM AND COAL TAR

Chemical or fraction	% by weight of petroleum tar (based on published figures)	Average % by weight of coke oven tar
Light oil	62.2	0.50
Carbolic oil	0 *	2.20
Tar bases	0	2.30
Naphthalene crystals } oil	7.4	11.9
Mono-methylnaphthalenes	3.1	1.6
Di-methylnaphthalenes	2.6	1.6
Acenaphthene fraction	0.3	1.5
Diphenylene oxide fraction	0	0.5
Fluorene fraction	0.2	1.0
Miscellaneous oils in the 'creosote' range	1.4	0.7
Anthracene—phenanthrene	4.6	4.7
Carbazole	0	2.0
Pitch (including pyrene, chrysene and miscellaneous chemicals usually included as anthracene oil)	18.2	69.5

for a relatively small proportion of the high-boiling coal tar fractions and that their production and marketing, however profitable, would not solve the problem of disposing of large quantities of oils, residues and pitches. However, there is no reason to suppose that the tar industry should not be able to imitate the petroleum industry and apply cracking processes, including high-pressure hydrogenation, to the high-boiling materials in an attempt to produce lower-boiling liquid fuels and perhaps light oil of the benzene type. Neutral middle oils from low-temperature carbonisation processes have been sold as diesel oil, indicating a possible outlet for residual oils from a tar chemical scheme. Production of stable and clarified high-boiling solvents by earth treatment is another possibility, especially in the case of bulked poly methylnaphthalenes. Pitch is still a useful binder in briquetting processes and the manufacture of lump fuel from small coal, but other possibilities lie in the manufacture of electrode coke and carbon ceramics.

A new outlook on tar processing

If only we could become serious about fully exploiting the potentialities of coal tar as a source of aromatic chemicals, in addition to solvents and liquid fuels, it might be possible to make a rational approach along the following lines:

(a) To separate and centralise as far as possible the refining of coke oven and other coal tars.

(b) To study in detail the effect of the nature of the parent coal, retort design and carbonisation temperature on the quantitative spread of chemicals throughout tars produced by carbonisation. It might eventually become possible to predict the type of coal and coal tar necessary to produce a given range of chemicals and oils of specified properties.

(c) To increase the efficiency of fractional distillation methods at present applied to the refining of coal tar and to substitute, wherever possible, batchwise distillation by continuous distillation.

(d) To apply to tar, prior to distillation, primary processes of molecular alteration, e.g. mild hydrogenation, so as to produce larger quantities of the readily utilisable, stable aromatics by distillation processes.

(e) To apply to distillation oils and residues, processes of cracking and solvent extraction so as to produce competitive liquid fuels of the diesel oil and benzole types.

(f) To bring all the techniques of organic chemistry to bear upon the finding of outlets for pure chemicals and crude distillation cuts.

The need for chemicals has been established. The petroleum industry has set about fulfilling it, at least in part, and the coal tar industry should be prepared to make its most effective contribution.

of the polymer, using the correlation rules established for simple substances.

The possibility of the presence of interpolymers based on two or more monomers must be considered, especially as in some cases the spectra of interpolymers may differ significantly from the sum of the spectra of simple polymers derived from the same monomers.

The identification of other constituents of polymer compositions such as plasticisers and lubricants is also possible by the infra-red method; since, however, mixtures may be encountered which contain one or more of a closely related series of compounds such as dibutyl, dihexyl and dioctyl phthalate, complete identifications may not be possible. In such cases valuable information is usually forthcoming from the examination of breakdown products such as alcohols, cresols and acids obtained from such a mixture. Examples were given of the way in which the constituents of the original composition may be deduced from an examination of this sort, using the infra-red technique in conjunction with established chemical methods.

Petroleum industry

Dr. H. Powell, discussing infra-red spectrometry in the petroleum industry, said that the method is extremely useful in the field of hydrocarbon gas analysis, as it makes possible the determination of the individual paraffins and olefines in a particular fraction, so long as the number of components present is not too large. It may also be used for the determination of carbon monoxide, carbon dioxide and sulphur dioxide in flue gases.

By suitable fractionation and infra-red examination of the fractions it is possible to determine the individual paraffins, naphthenes and aromatics boiling up to about 130°C. The number of olefines boiling above 50°C. is so large as to make a complete individual analysis impracticable. However, it is possible to estimate the amounts of the various types of olefine present.

Above 130°C. it is impossible to analyse any fraction completely, owing to the large number of isomers and the close similarity of their spectra. However, a certain amount of information can be obtained; for example, total aromatic content and olefine type analysis.

Infra-red spectrometry has also a general application to the numerous research and development problems in the petroleum industry.

Methods of obtaining a continuous examination of gas and liquid streams were described. These include the presentation of a portion of the infra-red spectrum on a cathode ray tube and continuous analysers in which filters are used to select the appropriate wavelength range.

Dr. Powell then gave a short account of structural analysis as an aid to the organic chemist and the possible applications of infra-red methods in the field of petrochemicals.

Infra-red Analysis

THE enormous expansion during the last five years of the application of infra-red spectroscopic methods to problems of analysis in the field of organic chemistry has led to much accumulated experience. As a result, it is now possible to assess on broad general lines the particular range of applicability of the infra-red techniques in relation to other methods of analysis. With this in mind, Dr. N. Sheppard discussed the scope of infra-red analysis at a recent meeting of the Physical Methods Group of the Society of Public Analysts. This was the first of three papers dealing with various aspects of the technique.

According to Dr. Sheppard, for the purposes of analysis, the most important criterion is that a particular substance or group should have at least one absorption band which is strong in comparison with the others in the same region of the spectrum. Whether or not suitable absorption bands are present can usually be settled by purely empirical means, but if there is considerable pressure on the time available on a given spectrometer it is of considerable use to be able to pre-select problems in terms of whether or not the infra-red method is likely to be of use. He then gave a short account of the main factors affecting the position and intensity of infra-red absorption bands and deduced some

generalisations as to the types of molecules or groupings that can most easily be detected or accurately estimated by infra-red methods. Some important apparent exceptions to these rules were also noted. The range of usefulness of the infra-red method as a whole was discussed in relation to other spectroscopic techniques, and some applications of the infra-red method to inorganic analysis were mentioned.

Polymer composition

The application of infra-red spectroscopy to the identification of organic materials was described briefly by H. A. Willis; simple mixtures may be analysed, since the spectrum of such a mixture is the sum of the spectra of the constituents.

In the case of polymer compositions, these are often mixtures of such a complex nature that extraction of single components by a chemical method is desirable before infra-red examination is attempted.

The infra-red examination of polymers extracted from compositions was described. In the case of simple polymers derived from one monomer, identification is easy, by matching against standard spectra of known polymers. When a new polymer is encountered of which the spectrum has not been recorded, valuable information may often be obtained from the infra-red spectrum concerning the chemical nature

Grangemouth Refinery Expansion

THE Grangemouth petroleum refinery on the Firth of Forth, operated by Scottish Oils Ltd., a member of the Anglo-Iranian group, is being enlarged with the object of raising capacity to 1,800,000 tons p.a. of crude oil, about four times its pre-war capacity. In connection with this development, a power station is being built. Another allied project is the building of a petroleum chemicals plant. According to a report on the progress of these schemes by A. Trystan Edwards, which appeared in a recent issue of the *Financial Times*, constructional work on the project began early in 1949 and the labour force required has been greatly increased since then to a current total of about 2,000 persons.

The project covers 350 acres and is being carried out in two stages, the first of which was completed last year. Major items of plant included in this first stage were a crude oil topping unit, a phosphoric acid treating plant and a copper chloride treating plant. Twenty-seven floating-roof storage tanks, 144 ft. in diam. by 30 ft. high and two, 116 by 30ft. are being added.

The new power station will have four boilers, each with a capacity of 150,000 lb./hr. at 500 lb./sq.in. pressure, and three 6,250 kW turbo generators will provide electricity. For cooling water there are two reinforced concrete cooling towers each with a capacity of 1,500,000 gal./hr. Make-up water is being provided by a joint scheme developed by the Grangemouth Burgh Council and the Stirlingshire and Falkirk Water Board.

Stage two is to be complete by mid-1952, and will provide for premium-grade spirits and special products. It will include a catalytic cracking plant, a vacuum distillation unit to prepare its feed-stock and a catalytic polymerisation plant to produce a high-octane petroleum from the light gases which are a by-product of the catalytic cracker. The main object of these two units is to turn low-grade fuel into high-grade motor spirit.

Additional storage tanks, despatching lines and pumps, and larger road- and rail-car filling sheds have been planned to meet the increased inward and outward volume of traffic. To accommodate the administrative and chemical staffs, new office and laboratory blocks are being erected. These will be sited one on either side of the entrance gate and so designed as to match architecturally the power station building, which will line up to the same frontage. Provision is also being made for additional housing in the neighbourhood to accommodate the increased number of employees.

Engineering problems

Constructional work at Grangemouth has brought up some interesting engineering problems. The south bank of the Forth is a layer of silty river mud, with
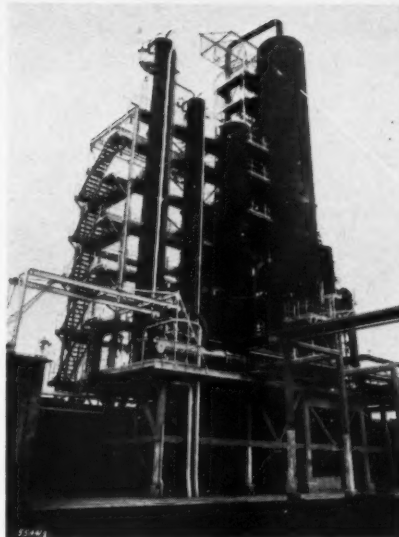
Crude oil topping unit.
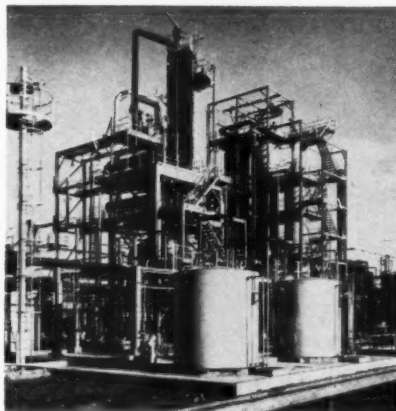
Phosphoric acid plant.
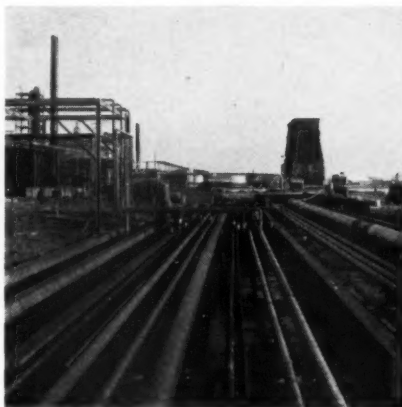
Main pipe track.

(All photographs by courtesy of Anglo-Iranian Oil Co. Ltd.)

a firm boulder bed lying at a depth of 120 ft. The existing plant was built on concrete rafts large enough to ensure that the load would not cause sinking, and friction piles were included where necessary. But the much heavier equipment of the new project ruled out this form of foundation and, as the cost of sinking piles to so great a depth would have been excessive, the buoyancy 'egg-box' type was selected. This consists of a hollow reinforced concrete raft sub-divided into compartments or cells.

The vertical panels are pre-cast and assembled on site, the reinforcing bars being welded and all junctions grouted up. Earth is then excavated from the compartments in such a way that the whole sinks evenly under its own weight. When the proper depth is reached, concrete bottoms are cast *in situ* and, finally, tops are added, making a complete series of air-filled watertight cells. Unexpected leaks can be detected and pumped out and, to ensure that no pockets of moisture remain below and that the soil has the maximum compactness, the method of electro-osmosis invented by L. Casagrande is being employed. The inventor, who is on the staff of the Building Research Centre, Watford, personally supervised this first large-scale use of his method in Britain.

Also of constructional interest was the erection of the 76-ton main fractionating column. This unit was fabricated to the design of the M. W. Kellogg Co. and moved by road in its entirety. On arrival at site it was erected in its vertical position, with base 20 ft. above ground level, in only 3 hr.

An essential element in the scheme is the development of shipping facilities. The two existing jetties at Grangemouth Docks, by which tankers have hitherto discharged their cargoes, will in the future be used for outward despatches of finished products. Incoming supplies of crude oil will be drawn from a new depot now under construction at Finnart, on Loch Long, 60 miles distant. This terminal will be connected with the refinery by a 12-in. all-welded pipeline, along which the oil will be pumped by direct-driven ram pumps, later to be replaced by electrically driven centrifugal pumps.

Radioactive pipeline cleaner

A noteworthy feature of this pipeline will be the use of radioactive material. In order to keep the interior of the line clean it is necessary that a scraper (called a 'go-devil') should be introduced periodically into the line and forced through the pumps. The use of a radioactive substance attached to the 'go-devil' will enable its progress to be followed through the line by means of a Geiger counter. The new Finnart depot will be able to accommodate large

CRYSTALLISATION

New crystallisers, salt production, recent installations, crystal formation

By A. W. Bamforth

THE process of crystallisation is one of the oldest known methods for the separation and purification of compounds, and when properly applied is often the best method for providing an attractive marketable product in a pure concentrated form.

Phase separation and recrystallisation

The use of graphical methods in phase separation by crystallisation is discussed by Newitt,[1] who gives examples of plots of mass of solute per unit mass of solvent against total mass of solvent.

Tipson[2] discusses the theory, scope and methods of recrystallisation to organic compounds. For purification involving a small portion of impurity a number of simple recrystallisations may be performed. The mother liquor is discarded or re-worked. In such problems the yield and degree of purification of the product for each cycle is important, and maximum yield per cycle is not always economical. In an example giving the successive yields on repeatedly recrystallised material, 13 recrystallisations at 95% yield per cycle give about the same final yield as six recrystallisations at 90% yield per cycle.

To avoid losses entailed by discarding the mother liquor, the principle of cascade or triangular fractional crystallisation is employed. This differs from simple recrystallisation in that both crystals and mother liquor are repeatedly fractionated. In the separation of two components A and B to obtain each substantially pure, this process or continuous crystallisation is essential.

A Swedish patent[3] deals with the separation of sodium from potassium chloride by treating a solid mixture of these salts with a solution of calcium chloride. Alternatively, brine containing both sodium and potassium is treated with solid calcium chloride. A double salt of calcium and potassium chloride is formed, leaving the NaCl behind as a solid. The double salt is concentrated and precipitated in a solid form and is stirred with an unsaturated solution of the double salt at a temperature below 37°C., whereupon solid KCl separates.

A further patent of Hampel and Norem[4] covers the separation of potassium chlorite and sodium chlorite from solutions formed
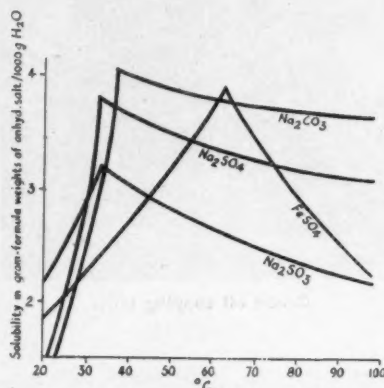
Fig. I.   Examples of salts having ' inverted ' solubilities.

by the interaction of ClO_2 with equivalent amounts of potassium and caustic soda. The solution is first evaporated to crystallise out the bulk of potassium chlorite. Further potassium chlorite is precipitated when the solutions are cooled to 25°C. The resultant mother liquor is seeded with sodium chlorite crystals and this salt crystallises out. The anhydrous sodium
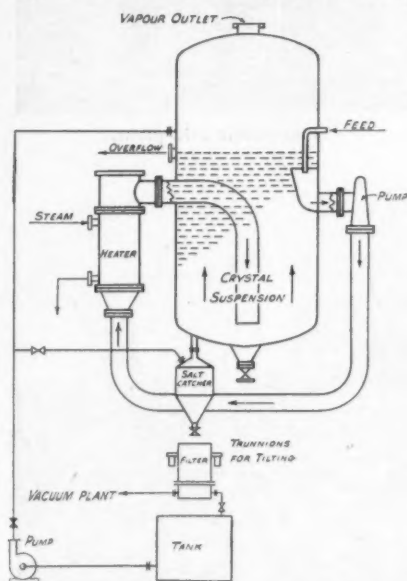
Fig. 2.   Salts which have the characteristic of decreasing solubility with increasing temperature, of which gypsum is a good example, are treated in an evaporator of this type.

salt is recovered by slurrying the hydrate with water at 38°C. and cooling the saturated solution to 25°C., whereupon the anhydrous salt crystallises out.

Types of crystallisers

Since supersaturation is an important prerequisite of crystallisation, Thompson[5] classifies crystallisers according to the method by which supersaturation is brought about or released. The classification covers supersaturation by cooling, by evaporation of the solvent, by adiabatic evaporation, circulation of solution over the crystal bed and salting out. He describes such well-known types as the Svenson-Walker, Wulff Bock, Lafeuille, Swenson, Krystal and Zaremba crystallisers, as well as the so-called standard types.

Few new types of crystallisers and crystallising methods have been reported since the last review.[6] An Italian patent[7] describes a crystalliser which takes the form of a rotating tube fitted with transverse perforated baffles. Saturated solution is introduced at the periphery and is distributed by the baffles as a mist which is cooled and crystallised by a current of air flowing through the centre of the tube. The crystals produced are in the form of a fine powder.

The boundary between the metastable and labile field of supersaturation is not normally sharply defined, and it is well known that rapid cooling of saturated solutions normally gives rise to small crystals of uneven size consisting of new nuclei and partly grown crystals. It is also true that small crystals and nuclei have a greater rate of solution than larger crystals. A Dutch patent[8] utilises both these facts in a process which is said to produce coarse uniform crystals notwithstanding rapid cooling.

Fine crystals and crystal nuclei which are formed by rapid cooling of a saturated solution at the externally cooled wall of a bath are conducted by a current generated by a stirrer into a warmer inner zone. In this zone area, the solution is undersaturated by reason of the rise in temperature, and the nuclei and smallest of the crystals are dissolved. The remaining crystals are then carried by the stream back to the cold wall, where, by cooling, the solution is again supersaturated. Further precipitation of salt occurs, partly as growth on the existing crystals and partly as new nuclei. These in turn are returned to the warmer unsaturated zone, where the second crop of small crystals and nuclei are dissolved. The cycle can be repeated indefinitely until the largest crystals are grown to the required size.

Production of various salts

The rate of production of a cooling crystalliser, without consideration of uniformity or size of the product, depends upon the rate at which the hot saturated solution can be cooled.

Martinez[9] gives a method of calculating the final crystallisation temperature to give maximum production for crystallisation processes which are based on temperature differentials. Equations and an example in the case of ferrous sulphate are given for batch crystallisation by natural cooling and similar equations are derived for crystallisation by artificial cooling in batch or continuous crystallisers.

For batch operation with natural cooling, yield per unit volume is plotted as a function of final temperature. From another equation the number of crystallisations per unit time are calculated and plotted as a function of the final temperature. The product of the ordinates of these two curves is plotted to give a curve of production per unit time against temperature. The resulting curve shows the maximum temperature which is the optimum temperature for maximum production per unit. For ferrous sulphate heptahydrate given in the example, the maximum optimum temperature obtained is $35°C$.

Ammonium sulphate is made from coke oven gas by the absorption of ammonia in sulphuric acid. Otto[10] describes a process where the effluent gas is subjected to mechanical separation to remove most of the entrained solution. An ammoniacal condensate from a subsequent operation on the gas is sprayed into the effluent, condensing an ammoniacal solution from the gaseous effluent from the spraying step and returning the ammoniacal condensate to the spray.

The preparation of magnesium, zinc, sodium, potassium or ammonium permanganate are described by Jaskowiak.[11] A solution of $Al(MnO_4)_3$ is prepared by the interaction of $KMnO_4$ and $Al_2(SO_4)_2 18H_2O$ in aqueous solution. Potassium alum formed at the same time is separated as crystals after cooling. When zinc or magnesium oxide, hydroxide or carbonate, alkaline earth oxide or alkali metal or ammonium oxide is added to the $Al(MnO_4)_3$ solutions the corresponding permanganate is formed, which may be recovered by recrystallisation from the supernatant liquor. The reaction temperatures are critical and vary in each case.

The preparation of sodium hydrosulphite is described by MacMullen.[12] Sulphur dioxide is absorbed in sodium sulphite at a rate to maintain a pH of 5 to 7. The resulting solution is contacted with sodium amalgam in a reactor and the concentration of hydrosulphite allowed to build up until crystals of $Na_2S_2O_4 2H_2O$ are formed. These crystals are removed more or less continuously and separated from the mother liquor in a filter press. The water of crystallisation is driven off by heating the dihydrate rapidly to $60°C$., after which the anhydrous salt is washed in alcohol and dried under vacuum. Yield is said to be in the order of 70%. The product is slightly contaminated with mercury up to 0.002%, but this is not detrimental for most trade purposes.
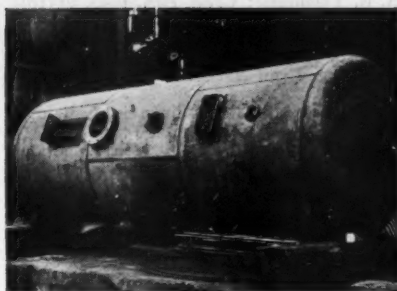
Fig. 3. Krystal evaporator designed for the production of 6 tons/day of anhydrous sodium sulphate crystals. The vessel is 5 ft. 6 in. diam. × 20 ft. high and is fabricated throughout in stainless steel.
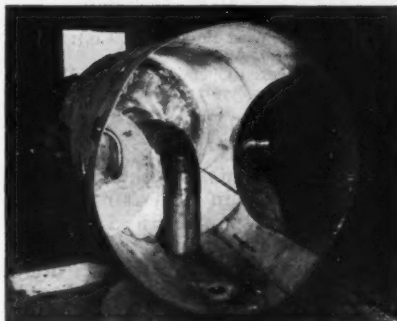
Fig. 4. Interior view of the modified Krystal evaporator shown in Fig. 2 designed for salts which have the characteristic of decreasing solubility with increasing temperature. Clearly visible are the central downcomer, the pump suction bowl on the right, and overflow on the left.

(*Photos : Power Gas Corporation Ltd.*)

What is claimed to be an economical and commercially feasible process for the manufacture of diammonium phosphate has been patented by Monsanto,[13] where $NH_4H_2PO_4$ crystals and anhydrous ammonia are introduced continuously into a saturated solution of ammonium phosphate $(NH_4)_2HPO_4$ contained in a closed reaction vessel. The reaction is carried out between 70 to $90°C$. and the pH of the solution maintained between 6.8 and 7.8. The $(NH_4)_2HPO_4$ crystals produced are mixed continuously with recycled mother liquor and separated by centrifuging. After drying, their basis alkalinity is 98% and apparent density 0.86 to 8.09 g./cc.

Gilpin[14] describes the crystallisation of magnesium hydroxide. Water containing convertible magnesium salts is treated with burnt lime in a reaction vessel. The liquid containing precipitated $Mg(OH)_2$ in suspension is transferred to a settling vessel. Settled seed crystals of $Mg(OH)_2$ are then returned to the first vessel until their weight is about 10 to 15 times the weight precipitated per hour. On filtering, cold cake containing 40% crystalline $Mg(OH)_2$ is obtained.

Production of salts with scaling tendencies

Vener and Thompson[15] have carried out work aimed at reducing the scaling ten-

dency of anhydrous sodium sulphate. The chief difficulties encountered with Na_2SO_4 are apparent from a study of the solubility curve. In the range of temperature from 32.4 to $125°C$. the anhydrous salt is in the stable solid phase, and its solubility in water decreases with an increase in temperature. From 125 to $234°C$. the solubility of the anhydrous salt increases with temperature, and beyond this point solubility again decreases with temperature.

With most types of crystallising and evaporating equipment a hard scale of the anhydrous salt forms on heating surfaces when the aqueous solution is evaporated. A new method proposed by Vener and Thompson[16] involves the controlled feed of a concentrated aqueous solution of Na_2SO_4 into an evaporator containing an organic liquid which is miscible with, but less volatile than the water. A marked lowering of the solubility of the anhydrous salt is evident, due to the salting-out effect of the organic solvent and, as previously reported,[17] the presence of ethylene glycol in aqueous solutions of Na_2SO_4 makes the solubility almost invariant with temperature.

Wiseman, Blackman and Hellmer[18] describe a specially designed evaporator heated with flue gases, which is said to be particularly suitable for the concentration of anhydrous sodium sulphate.

A modified form of Krystal evaporator crystalliser[19] is especially suitable for the crystallisation of salts of inverted solubility, for, by control of the supersaturation, scaling of heating surfaces is eliminated. Figs. 2, 3 and 4 show an example of this type designed by the Power-Gas Corporation Ltd., of Stockton-on-Tees, for the production of 6 tons/day of anhydrous sodium sulphite, a salt having similar characteristics to anhydrous sodium sulphate.

New installations

An installation comprising Swenson evaporators for the recovery of hydrated sodium sulphate and concentration of sulphuric acid from rayon spin bath solution has been described.[20] This plant is for the production of 4,000 lb./hr. of crystalline material.

The installation of a new Krystal evaporator in the Consolidated Mining & Smelting Corporation's plant in Canada, whose metallurgical and chemical operations are the most modern of their kind in the world, has been reported.[21] The unit is constructed mainly of stainless steel and is the last of five such units installed for the production of ammonium sulphate.

A comprehensive scheme of plant development at the Kembla works of Australian Iron & Steel Ltd. includes the installation of a Willputte by-product ammonium sulphate plant including Krystal crystallisers. The benefits which can be obtained from this plant are enumerated and the plant itself described in some detail.[22] A further Krystal evaporator installation for the recovery of ammonium

sulphate from coke oven gas at Hamilton, Ontario, has also been described.[23]

Bell[24] reviews ammonium sulphate production methods and describes direct, indirect and semi-direct processes. Kowalski[25] gives a technical review of potash production in various countries. Hamm and Avery[26] describe the solar salt industry of the Philippines which produces an inferior salt contaminated with calcium and magnesium sulphates. The Californian solar salt production technique[27] was investigated and it was found that concentrating the brine to 25°Be before passing it to the crystallising tanks increases the efficiency and may lower operational costs. During the investigation it was found that the depth of pond has no significant effect on evaporation, but evaporation is more rapid if the bottom of the crystallisation tank reflects the sun's rays.

A review with 35 references in the preparation, properties and uses of pyrophosphates, polyphosphates and potassium and sodium metaphosphates (both glass and crystals) is given by Garre and Huber.[28]

Change of crystal habit

Borax has a very strong effect on the crystallisation of epsonite. The prismatic habit of crystals grown from pure solutions is immediately changed to the tetrahedral by the addition of 1% borax. Mokieskii and Mokievskaya[29] have studied the effect of borax additions ranging from 0.01 to 5% borax.

Frejacques[30] reports that the addition of 0.2 to 0.5% of a solution of cyanic acid or cyanates to a solution of urea causes this salt to crystallise in a sandy form.

Photographs published by Vener and Thompson[15] serve to emphasise the remarkable change in crystal structure which can be made by the addition of an impurity or an alteration in the technique of crystallisation. Normal anhydrous sodium sulphate crystallises as hard cubes. Crystals precipitated from aqueous solutions by the addition of 60 to 70% glycol without stirring are light and fluffy, similar to glass wool in appearance, whereas crystals precipitated from solutions as above, but vigorously stirred, are well defined.

Rohner and Wood[31] have patented a process of coating granular ammonium nitrate with 200-mesh urea formaldehyde resin. Ordonneau[32] has studied the hygroscopicity of potassium chloride in relation to its caking tendency. Treatment of the crystals with 1% of high-boiling petroleum is said to suppress caking tendencies almost completely. The crystal size and state of the surface of the faces affects the caking tendency. Uniform, well-formed crystals are less susceptible to caking while in storage.

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Acid handling

In the foreword to this book* it is stated that the most modern methods now practised in the works are described in order to assist those who are less experienced. ' If these methods are strictly adhered to, human accidents and plant damage should be little greater than those experienced in dealing with non-corrosive liquids.' The Ministry of Supply memorandum on ' The Transport and Handling of Nitric and Sulphuric Acids ' and a booklet issued by the Pennsylvania Salt Manf. Co. on hydrofluoric acid have both been consulted.

The layout of the book is indicated by the chapter headings: Materials of construction; storage of acids; transport in containers; pipe lines; valves and cocks; pumps; accident prevention; battery acid; anhydrous hydrofluoric acid.

The book is most comprehensive, although rubber is omitted as a material of construction.

Photographs of tanks are rarely useful and the photographs of plug cocks, and globe, plug, diaphragm and gate valves presuppose a lack of elementary knowledge on the reader's part, especially as the chapter on pumps tends in the opposite direction. The vector diagram for centrifugal pumps will not be of much use as it stands. It is also unsafe to give dimensions for all the variables in the Reynolds number with the exception of density, for a reader who has not even been told that it is dimensionless. The use of a complicated formula for turbulent flow is not justified in commercial rough pipes where the friction factor is independent of the Reynolds number. The method of designing pipe lines based on average commercial practice, under a given set of conditions not fully specified, reminds one of the adage that a little knowledge is a dangerous thing.

Acid Handling: The Transport and Handling of Sulphuric and Hydrofluoric Acids. Imperial Smelting Corp. Ltd., pp. 68, 1950, 10s. 6d.

The corporation are to be congratulated on bringing out a handbook which will certainly be of considerable help in acid handling, especially with regard to safety precautions.

M. B. DONALD, M.I.CHEM.E., F.R.I.C.

Grangemouth Refinery Expansion

tankers of 28,000 tons deadweight, of which the British Tanker Co. (Anglo-Iranian shipping organisation) at present has on order.

Petroleum chemicals

A project closely allied to that of Grangemouth is the British Petroleum Chemicals plant, which will adjoin the oil refinery. The company was formed in 1947 with £5,000,000 of capital jointly held by Anglo-Iranian and the Distillers Co. It will draw its raw material mainly from the refinery, and will also derive its steam and electric power from the latter. Here, too, construction work is well advanced. The plant will initially produce ethyl alcohol and iso-propyl alcohol, and other petroleum chemicals at a later stage. The products will be consumed in the plastics and rayon industries and for drugs, dyestuffs and textile manufacture.

British Petroleum Chemicals has also recently formed with Monsanto Chemicals Ltd., a joint subsidiary, Forth Chemicals Ltd., for the manufacture of monomeric styrene for the plastics industry. It is probable that this will necessitate the construction of a further, though smaller, plant at Grangemouth.

The cost of the Grangemouth refinery expansion and British Petroleum Chemicals projects were together estimated at £12,000,000. Having regard to the rising trend in refinery costs and particularly the dollar element since devaluation, this can probably be regarded as a conservative estimate.

Production of Caustic Soda and Chlorine at Point-of-use

Plants for the manufacture of caustic soda and chlorine in the small quantities needed in many industries have been designed by an American firm. Such units, it is claimed, can produce each 24-hr. day some 225 lb. of 100% caustic in 50% solution, 200 lb. of chlorine and 5 lb. of hydrogen, from 350 lb. of salt and 40 gal. of water. Here is a detailed description of these new plants.
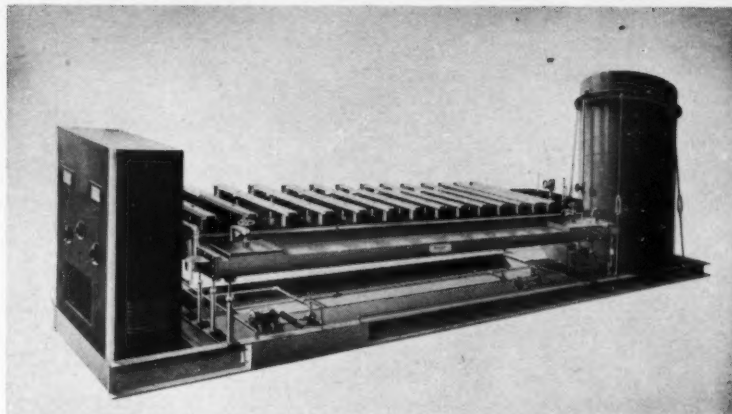
Packet plant for production of caustic soda and chlorine.

EFFICENT production of caustic soda and chlorine, in the quantities required, at each point of use, has heretofore been largely impractical. Up to now the heavy capital expenditures involved have required production of these basic chemicals at the few points where low marketing, raw material and power costs converged. Consumption, on the other hand, takes place in a multitude of widely scattered points and the quantities used are, in most cases, microscopic by comparison with the output of the major producers.

Units for the production of caustic soda and chlorine at point-of-use have been designed and constructed by Amroc Inc. of New York. One of many such units is known as the packet plant. When delivered, the purchaser need only remove the crating, connect the available utilities, put in the mercury (included, but shipped separately) and then fill the brine saturation tank with common salt. He then has a complete caustic-chlorine producing machine in operation which, it is claimed, requires less than 1 man-hr./24-hr. day for continuous operation and servicing.

This plant occupies a floor space of only 25 by $3\frac{1}{2}$ ft. Its guaranteed minimum output per 24-hr. day is 225 lb. (102 kg.) of 100% caustic soda, 200 lb. (91 kg.) of chlorine and 5 lb. (2.3 kg.) of hydrogen. The caustic soda is the high-quality salt-free rayon grade type. The chlorine and hydrogen are more than 98% pure and almost entirely free of carbon dioxide and mercury vapours. From these raw materials over 130 gal. of 15% hypochlorite or 600 lb. of 21° Baume hydrochloric (muriatic) acid can be produced. The equipment for the production of either of these derivative chemicals can be mounted on the same single (25 by $3\frac{1}{2}$ ft.) base upon which the packet plant is assembled and delivered.

The requirements for this output in raw materials are 350 lb. of salt, 40 gal. of water and 425 kWh of electricity; and the raw materials (salt, water and electric power) which are available locally are practically always highly suitable for use.

Operation

The flow process of the plant, as well as larger Amroc units, is as simple and streamlined as the appearance of the plant itself (see Figs. 1 and 2). Three streams enter—water, salt and rectified power. In the heart of the cell, a simplified trough-type electrolyser, the salt-saturated brine is converted into chlorine at the graphite anode and into sodium metal at the flowing mercury cathode. The sodium metal forms an amalgam with the cathodic mercury and is thus carried to the denuder section. Here it reacts with counter-current flowing water to form hydrogen gas and caustic soda, the latter in any concentration desired up to 50%. The overall and sectional reactions may be represented as follows:

Overall:
$$2NaCl + 2H_2O + Power \rightarrow 2NaOH + H_2 + Cl_2$$

Electrolytic cell:
$$2NaCl \rightleftharpoons 2Na^+ + 2Cl^-$$
$$2Na^+ + 2e \rightarrow 2Na$$
$$2Cl^- - 2e \rightarrow Cl_2$$
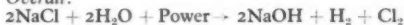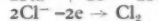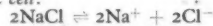
Denuder:
$$2Na + 2H_2O \rightarrow 2NaOH + H_2 \uparrow$$
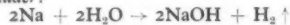
The denuded mercury is returned to the electrolyser section for further service and the depleted brine is re-saturated for re-use. Chlorine, caustic and hydrogen products are used as such or are fed to the hypochlorite tower or hydrochloric acid units. All these operations are carried through almost entirely automatically.

The small producer of caustic and chlorine needs a plant with the following general specifications: (1) Low first cost and depreciation rate; (2) economy and flexibility of operation; (3) low labour, supervision and maintenance charges; and (4) high-purity products.

It would be impossibly costly for each user to obtain the skilled engineering to develop such a plant. But the engineering charge has been spread over many units with a consequent reduction in development expense to users of the units. This contributes to the initial low capital expenditure.

A further factor is that the plant is shipped as a complete unit, including everything necessary for flexible production and economical maintenance over a long life. Extensive auxiliary utility installations are eliminated by providing a single bus-bar connection, factory-assembled piping and one standardised motor for circulation of all moving materials.

The use of durable materials such as rubber-lined steel, corrosion-resistant piping and specially designed graphite anodes assures a good working life. Low maintenance material costs are effected by the use of standardised parts throughout. All materials are claimed to be resistant to both physical and thermal shock. No concrete or ceramic is used whatsoever. Because of the quality of the equipment provided, a plant amortisation rate of 5% p.a. is conservative. Details of the plant are as follows.

Power

Each plant is designed to function with the available power supply. No modification is usually required, so far as the mercury, brine and caustic circulation motor is concerned. For the electrolytic process a selenium-type rectifier with a power control board, as well as cell cut-out device, which protects the installation in case of power failure, is provided. This converts the A.C. current into the 4-V D.C. current required for the cell.

Where electric power is not available, irregular in supply or high in price, or where excess steam or D.C. power is

available, a power plant designed to meet these special situations is included.

The plant cell operates at 3,000 amp. and an average of 4 V. Current efficiency is 94 to 96%. Power consumption is said to be less than 425 kWh/24-hr. day for the entire plant; this includes both the electrolytic process and motor operation power requirements.

The plants may be operated at from 20 to 120% of their rated capacity without damage to the quality of the products, the cell itself or without decreasing the current efficiency. As a consequence, production may be scaled inversely to varying raw materials, labour and power costs, while still meeting the needs for the products. Specifically, when power charges are based on a 'maximum demand' schedule, top production may be scheduled for times when such demand is at a minimum.

Salt and water supply

Either common rock salt or sea salt may be used. The plant operates with a minimum of purification equipment. This necessitates the periodic flushing away of accumulated impurities. The elimination of operations and special salt purification chemicals more than compensates for the slight loss of salt in this operation.

Almost any available water supply may be used, provided that small quantities of caustic soda and barium chloride or carbonate are added once daily to the brine re-saturation tanks.

Total water consumption (when making 50% caustic) is approximately 70 gal. daily; 350 lb. (750 kg.) of salt are used and, for brine purification, small amounts of caustic soda (as made by the plant itself) and barium carbonate or barium chloride.

Automatic re-saturation, re-circulation and purification of the salt solution means that the salt and negligible amounts of purifying chemicals need be added but once during each 24-hr. day. The make-up water value is adjusted at this time and (aside from an occasional flushing of the re-saturation tank), this is the total labour demand for this part of the plant.

Mercury and graphite

Mercury circulation, cleaning and replacement has been one of the largest sources of difficulty in previous amalgam cell operation. A simple but effective mercury pump and clearing device is an integral part of the plant. The entire process is carried out automatically and the replacement of mercury and of graphite anodes is estimated to cost less than $U.S. 15 per day's plant operation.

The skimming of solid impurities deposited upon the mercury is automatic and results in no cell operation interruption. Similarly, the amalgam denuder can be opened and inspected or cleaned when necessary without stopping production. Therefore, the mercury is always clean and the cell operates with consistent good results over long periods of time.
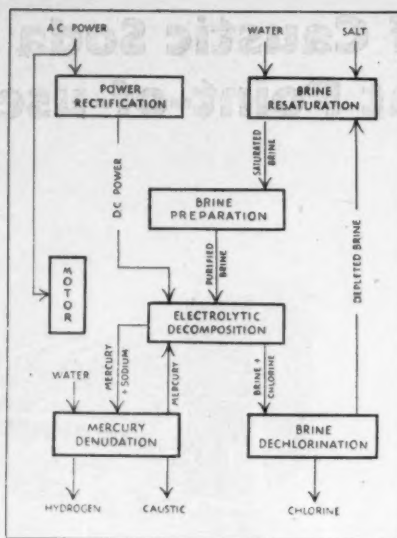
Flow diagram of packet plant, excluding derivative products.

Caustic soda

As mentioned, the caustic soda produced is the salt-free (rayon grade) type. The exact concentration (up to 50%) is controlled solely by the setting of one denuder water-supply valve and is continuously indicated by a built-in gauge. The resulting solution is a clear, water-white product ready for immediate use.

This eliminates both labour-consuming and dangerous caustic solution preparation, as well as the expensive steam evaporation and purification equipment necessary to convert many ordinary commercial caustics to the pure 50% solution yielded by the plant.

Chlorine and hydrogen

The chlorine is produced in a concentration of 98 to 99%, with an average of less than 0.5% carbon dioxide and less than 0.5% hydrogen. It is removed (by means of a built-in fan) at a negative gauge pressure. The high purity of the chlorine makes its liquefaction or conversion easy and safe. Losses in the available chlorine in bleaching powder and hypochlorite solutions, which invariably result during periods of storage, are largely

TABLE I. CAUSTIC SODA AND CHLORINE PACKET PLANT DAILY PRODUCTION SCHEDULE

Finished products:	
	lb.
100% caustic soda (in 50% solution)	225
Chlorine	200
Hydrogen	5
Raw materials:	
Salt	350 lb.
Water	40 gal.
Maintenance materials:	
Cost is less than $U.S.0.15 worth per day	
Power: 425 kWh	

eliminated. With local production, such products may be used while fresh, or may be prepared and kept in low concentration.

Great savings result from eliminating the return transportation of empty chlorine or hypochlorite containers. Where the chlorine is being consumed automatically (as for water purification or in preparing a hypochlorite solution) the plant may be operated during extended periods with no attendance.

The hydrogen is taken from the denuders at a gauge pressure up to 50 mm. water column, thus eliminating all possibility of air dilution. It is more than 99.5% pure, without trace of mercury vapour. Its high purity makes it ideal for the production of hydrochloric acid or oil and fat hydrogenation.

Auxiliary equipment available for operation with the plant includes that for liquefying chlorine, producing solid, flaked or C.P. caustic soda, caustic potash, a solution of calcium hypochlorite, or sodium hypochlorite, liquid chlorine, hydrochloric acid, carbon dioxide, calcium chloride, chlorinated hydrocarbons, etc. This equipment is built to the same specification as the plant. In many cases it can be installed upon the same single base as the plant.

Housing, installation, repair and maintenance

Installation is a simple operation. No special housing is required. Everything necessary for the entire plant is received already assembled upon a single base. No overhead crane or similar moving device need be employed. Uncrating and connection to utilities completes the installation.

A selection of repair parts and extra supply of graphite anodes comes with each plant. High-quality materials of construction eliminate costly shut-downs. An ordinary mechanic may take care of what little maintenance is necessary. All piping and current conductors are arranged so that they do not encroach upon the accessibility of the cells for operation, inspection or maintenance.

The only work of a special kind (which need be done only once every two or three years) consists of renewing graphite anodes. Anyone of average skill can do this by following the instructions supplied with each plant covering this operation.

Finishing process for stainless steel

A process for creating a smooth finish on stainless-steel spinning jets is described in a recent patent (S. A. Mansfield, U.S. Pat. 2,496,299). The spinning jets are subjected to electrolytic anodic action in a bath consisting of 40 to 50% orthophosphoric acid and 50 to 60% glycerin.

A current density of 0.1 to 2 amp./sq.in. is employed and the bath temperature is maintained between 40 and 125°C.

The Recovery of Benzole at Gasworks and at Coke Ovens

By (the late) W. H. Hoffert, M.A., B.Sc., F.R.I.C.,* and G. Claxton, M.Sc.

(National Benzole Association)

Here is a summary of one of the papers of chemical and chemical engineering importance which were delivered at the Fourth World Power Conference held in London during the summer. Developments in the oil-washing process are reviewed, some space being devoted to the problem of sludging. A plant for washing gas with oil under pressure is briefly described. Active-carbon recovery is outlined in the light of the modern, automatically-operated plant. Some mention is also made of recovery by cooling. Refining covers especially the removal of carbon disulphide and thiophen. The effect of the refining process on the corrosion of equipment and the production of corrosive substances and mercaptans in the refined benzole are described. Refining by hydrogenation methods is dealt with and, finally, a brief mention is made of the trends that might be expected in the recovery and refining of benzole.

Production at coke ovens

THE thermal efficiency of blast furnaces has steadily improved during recent years, and in America low-cost oxygen is now being used to improve the efficiency still further.[1] Thus, provided steel output remains constant, a fall in the demand for metallurgical coke should be anticipated, with a consequent fall in the output of benzole from coke ovens.

Production at gasworks

Prior to 1938 many of the smaller gasworks in Great Britain did not consider it profitable to recover benzole. Under today's unsettled conditions no useful purpose can be served by debating this polemical subject. Nevertheless, in 1938 it was estimated that the gas industry, as distinct from coke ovens, could contribute a further 20×10^6 Imp. gal. (91×10^9 kl.) of motor benzole p.a., equivalent to about 23×10^6 Imp. gal. (115×10^9 kl.) of crude benzole. In spite of this potential source of extra benzole, the production at gasworks only reached a maximum increase of 17×10^6 Imp. gal. (77×10^9 kl.), and for most of the period under review it increased by much less than 10×10^6 Imp. gal. (45×10^9 kl.). This was not due to any lack of enterprise, but to the increased demands for gas during the war years, shortage of coal, and stoppages due to bombing, as well as the limitations set by calorific value.

The outstanding difference between the gasworks and coke ovens as regards benzole recovery lies in the fact that extra benzole recovery at gasworks meant the installation of new plant at works that had previously not recovered benzole. According to the Ministry of Fuel and Power, 207 new recovery plants were installed at gasworks during the war.

Oil washing

There have been no outstanding new developments in the recovery of benzole by

*Mr. Hoffert died on August 1, 1949, at an early stage in the preparation of the paper.

oil washing in Great Britain since the erection of a vacuum-recovery plant, designed about 1934 by the Gas Light & Coke Co. in co-operation with W. C. Holmes & Co. Ltd.[2] About this time considerable attention was being paid by gas engineers to the reduction of the sulphur content of coal gas. Wood and Claydon[3] state that 10 gr./100 cu.ft. (0.229 g./cu.m.) have been recommended as the maximum permissible sulphur content in the Heyworth report,[4] but some authorities consider that 3 gr./100 cu.ft. (0.069 g./cu.m.) should be aimed at.

The vacuum plant referred to above circulates a quantity of oil equivalent to 35 to 40 Imp.gal./100 cu.ft. (54.7 to 62.5 kl./cu.m.) of gas washed at normal temperature, or about four times that normally employed for benzole recovery. By using this high rate of oil circulation, it is possible to remove 90% of the carbon disulphide in the gas or 75% of all the sulphur compounds. The corresponding removal of benzole is about 85% of the initial benzole content of the gas, but this value can be adjusted at will in the following manner. The vapours from the still are divided into three fractions, the lightest of which contains most of the sulphur compounds. The second or intermediate fraction consists of benzene, toluene, xylene and other hydrocarbons of similar boiling range, while the heaviest fraction contains naphthalene and heavy hydrocarbons, including those distilled from the wash oil. The intermediate fraction is substantially free from both naphthalene and organic sulphur compounds, and may thus be returned to the oil to effect any necessary adjustment in total benzole recovery.

The major items of costs for operating this plant are the extra steam and cooling water required. Oil-to-oil and vapour-to-oil heat exchangers are used, and distillation is carried out under a partial vacuum. The conditions are adjusted so that very little additional live steam is required in the process, exhaust steam from the vacuum pump and the turbines driving the

oil and water pump being practically sufficient to operate the plant.

Sludging of wash oil

Although at one time benzole was recovered from gas almost entirely by washing with creosote oil, the increasing recovery at gas works, where the removal of naphthalene from the gas is necessary, encouraged the use of petroleum oils as an absorbent for recovery, since this oil is originally free from naphthalene. From about 1930 onwards the rise in price of creosote, compared with petroleum oils, also encouraged the use of these oils at coke ovens.

During use, creosote thickens, whereas gas oil deposits an insoluble sludge, particularly in cooler parts of the recovery plant, such as oil coolers, thereby seriously impairing the efficient working of the plant. Attention had been drawn to this property of gas oil, and many papers on the subject have appeared. During the war, however, high efficiencies of recovery at coke ovens were being sought, and in Great Britain the Benzole Technical Committee investigated the sludging of petroleum oils used for benzole recovery. The findings of this committee were published in 1947.[5] The conclusions drawn by this committee indicate that there is no single cause to account for sludging. Since sludging is more prevalent at coke ovens than at gasworks there seems little doubt that hydrogen sulphide contributes to the trouble. It also seems likely that resinification of high-boiling unsaturated hydrocarbons removed from the gas, but not subsequently separated from the oil in the stripping process, is a frequent source of the sludge.

It is recommended that stripping of the oil should be as complete as possible, and that this stripping should be carried out at as low a temperature as practicable, by the use of open steam rather than by increase in the oil temperature. Pre-treatment of the gas by a small quantity of oil, which is

'recycled' in a separate system, is beneficial, and tar precipitators should be operated at as low a temperature as possible. Sludging troubles, but not sludge formation, may be reduced by providing points in the oil circulation system where sludge and emulsions have time to separate, and from which they can be drained. Contact of the debenzolised oil directly with water, for example by using direct oil coolers, was also found to be beneficial.

Oil washing under pressure*

The theoretical advantages of washing compressed gas with oil have long been recognised. Advantage can be taken of these benefits more readily when it is necessary to compress the gas for reasons not connected with benzole recovery. High-pressure gas distribution over comparatively long distances, at a pressure of about 30 lb./sq.in. (2.1 kg./sq.cm.) is now not unusual, and a plant has been erected, and is working successfully, at the works of the Hemsworth & United Kingdom Coke Oven Co. Ltd.

Two streams of crude gas are washed at this works, one at atmospheric pressure for supplying the coke ovens, and the other at 30 lb./sq.in. (2.1 kg./sq.cm.) gauge pressure for purification and distribution. Similar scrubbers, although of different dimensions, are used on both gas streams.

A higher efficiency of recovery was obtained in the high-pressure scrubbers, despite a smaller scrubbing area and, on the average, a lower rate of oil circulation.

Another considerable advantage of washing the gas under pressure is the increased removal of organic sulphur from the gas.

Active-carbon recovery

Benzole recovery by the use of active carbon was started on a commercial scale in Germany some 25 years ago. Following experiences with a 2,000,000 cu.ft./day plant at the South Harrow gasworks of the Gas Light & Coke Co., a plant capable of dealing with 75×10^6 cu.ft./day of gas was erected at Beckton for the same undertaking, and another plant has subsequently been erected at Bromley. The Beckton plant has been fully described at the Chemical Engineering Congress.†

Recent development has been in the direction of mechanising the operation of active-carbon plants.

A number of automatic active-carbon plants have been erected by Sutcliffe Speakman Ltd.[6] All these plants are operating on purified gas.

The plant diagrammatically illustrated in Fig. 1 consists of two cylindrical adsorbers containing a thin annular layer of active carbon held between stainless steel screens. Gas passes from the outside to the inside

*Information privately supplied by H. S. Illingworth.

†W. G. Adam and G. W. Anderson: 'The Recovery of Benzole from Coal Gas,' *Trans. Chem. Eng. Congress*, World Power Conference (London 1936), 2, p. 135.

of the annular layer, and steam in the reverse direction. The gas flow to each adsorber is controlled by three valves, which are of the mushroom type, diaphragm-operated by pneumatic pressure. The valves are spring-loaded so as to return to a position giving free flow of gas in case of failure. During the drying and cooling period a portion of the stripped and cooled outlet gas is recirculated through the appropriate adsorber by means of a fan, being slightly heated by passing through a small gilled steam heater before entering the adsorber. The time cycle is controlled by a cam system driven by an electric motor or governed steam engine, which also drives a compressor for operating the valves. The steaming and cooling periods are equal in length, and together equal the adsorbing period. A notched valve and indicator are provided so that the rate of steaming can be varied to alter the total steam admitted during the quarter cycle. It should be noted that only a portion of the stripped gas from one adsorber passes through the second.

In the Newton Chambers' plant (Fig. 2) there are two rectangular adsorbers of welded construction, each containing two thin vertical layers of carbon. Gas travels through the two layers in series, but during the steaming period steam from a perforated pipe between the layers passes steam outwards through the carbon. Thus, during steaming, steam passes through one layer of carbon in the same direction as the gas flow, and in the other layer contrary to the flow.

Two circular valves, divided into four sectors, contain a lubricated rotor attached to a diametrical division-plate, which turns through 90° when operated, through a ratchet mechanism, by means of a hydraulic cylinder operated by town's main pressure. A benzole condenser, separator and final gas-cooler are provided.

Operation of the time cycle is ingenious. A small quantity of waste water leaving the benzole condenser, governed by a constant head and fixed nozzle, flows into a closed tank fitted with a movable tube, which acts

as an air vent, and syphon discharge. When the water level reaches the end of the tube, no more air can leave the tank, and water is forced over the syphon into a counterbalanced bucket, which operates a valve directing water to one or other of the gas valves, and at the same time introduces steam to the appropriate adsorber. A small pump circulates cooling water from a convenient source. Only a small amount of town's water is used.

This plant differs from the one described above in one important respect. Stripped gas from one adsorber is passed, without cooling, through the newly-steamed adsorber in order to cool it. A plant to this design, with a maximum capacity of 250,000 cu.ft. (7,080 cu.m.)/day only, situated at Penistone, has been in operation for some time. More recently a plant for 4×10^6 cu.ft. (113×10^3 cu.m.)/day has been erected, and an installation of similar capacity together with a smaller one are in course of erection.

Recovery by cooling

From time to time during the years under review, papers have appeared recommending the recovery of benzole by cooling. Normal coal gas contains benzole at a partial pressure of about 7 mm. Hg. At 0°C. (32°F.) the gas will become saturated with benzole, and at -50°C. (-60°F.) the vapour pressure will be negligible and practically all the benzole will be removed. Complete removal can be achieved at a higher temperature by also compressing the gas. This latter method is that employed by the Société Linde and described by Berthelot,[7] who claims that, although the cost of recovery by cooling and compression is greater, a yield of practically 100% is possible, compared with a yield of only 87% by oil washing. Thus the total revenue might be greater.

In 1942 an experimental Dempster Mauiri plant was installed at the Droylsden works of the Manchester Corporation Gas Department.[8] This plant used the Mauiri system of ammonia refrigeration and
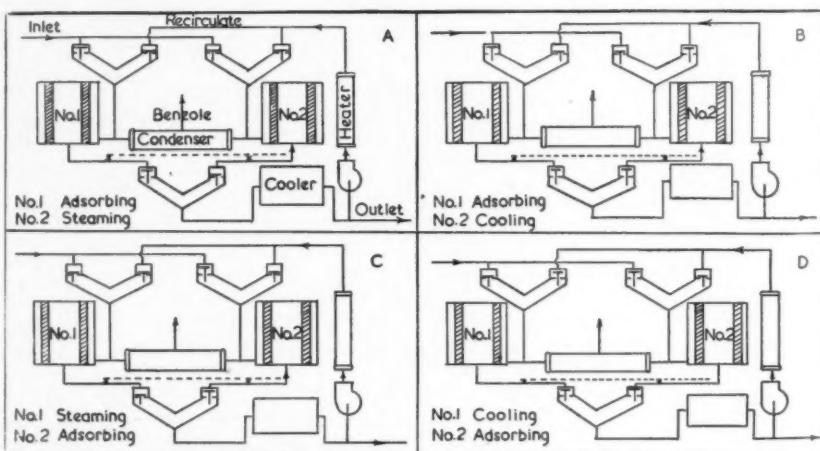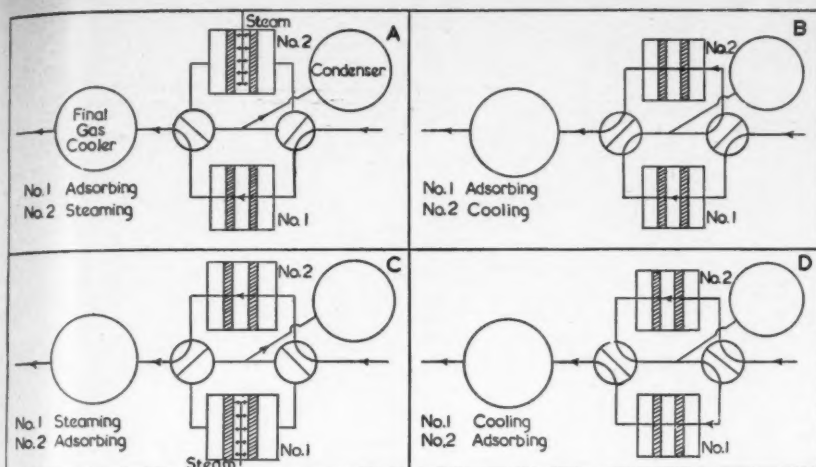
Fig. 1. Sutcliffe Speakman active-carbon plant.

Fig. 2. Newton Chambers' active-carbon plant.

cooled the gas to -40 to -50°C. (-40 to -60°F.) without compression. The plant showed that, although the refrigeration could be carried out efficiently and cheaply, considerable difficulties arose due to the deposition of solid benzole. The difficulties were not overcome by spraying or evaporating toluole into the gas, and the experiments were not continued.

While there may be some advantages in the recovery of benzole by freezing methods, particularly if compression of the gas is desired for the purposes of distribution, it is felt that experience, particularly in Great Britain, is at present insufficient to enable a sound assessment of the value of such processes to be made.

Refining

At the time of the Third World Power Conference, the production of motor benzole by the use of inhibitors had become firmly established in Great Britain, and was in operation on the continent of Europe.[9]

The process has been fully described on many occasions, but for the benefit of those who are not familiar with the benzole industry, it may be stated briefly that the inhibitor process consists of adding to freshly distilled benzoles, treated previously if necessary to remove colour and sulphur compounds but still containing most of the unsaturated hydrocarbons, small quantities of up to 0.05% by weight of oxidation inhibitors, in order to prevent the subsequent formation of high-boiling resins. These inhibitors were generally a special cut of cresylic acid, high in the o- and p-compounds, often mixed with catechol.

Before World War 2 the demand for benzene, toluene and xylene for chemical purposes was but a small proportion of the total production of crude benzole. The chief source of revenue from benzole, therefore, was as a motor fuel. The use of the inhibitor process enabled an average increase in the amount of saleable motor benzole of about 3% by volume to be made.

This new process did not achieve success without some attendant troubles, not the least of which was that of limiting the sulphur content of the refined material.

Sulphur removal

It may be said that the yield of saleable motor benzole obtainable from a given crude benzole depends upon its sulphur content, particularly upon its thiophen content. As is well known, the chief sulphur compounds in crude benzole are carbon disulphide and thiophen. These frequently exist in approximately equal proportions, but benzoles from active-carbon or vacuum-oil plants may contain a preponderance of carbon disulphide. Benzole recovered from vertical-retort gas also contains other sulphur compounds such as sulphides, disulphides and mercaptans.

(a) **Carbon disulphide removal**. The three chief methods of removing carbon disulphide, already fully described elsewhere, are:

(1) Fractionation.
(2) Methanol soda.
(3) Ammonium polysulphide.

(1) *Fractionation*. Removal of carbon disulphide by fractionation cannot be achieved without the simultaneous loss in the forerunnings fraction of other compounds, some of which have a fuel value. Cyclopentadiene will be present in this fraction to an extent depending not only on the quantity recovered from the gas, but also on the age of the crude benzole. Cyclopentadiene readily polymerises to its dimeric form which, on refractionation of the crude benzole, slowly depolymerises. Thus, cyclopentadiene may also be found in any fraction produced by distillation of crude benzoles containing the dimer. This, as will be shown later, is an important factor in treating crude benzoles with sulphuric acid.

Removal of carbon disulphide by ordinary equipment results in a comparatively large forerunnings fraction, which may, however, be treated subsequently by

the chemical methods described below for carbon disulphide removal.

Considerable use has been made in recent years of the Barbet column,[10] which, by using well-designed and efficient trays and bubble-caps, enables the whole of the carbon disulphide to be concentrated in a fraction of not more than twice the volume of the carbon disulphide present. In addition to this fraction, however, there is a further loss of 1 to 3%, and perhaps more, from the vent to the condenser. In order to prevent this loss, Midland Tar Distillers Ltd. have taken out two patents for an apparatus,[11] operating on their own works, for saving this loss. The uncondensed vapours are recovered continuously by absorption in light solvent (boiling point 140 to 160°C.; 285 to 320°F.), which is subsequently treated with ammonium polysulphide to remove the carbon disulphide. This process is reported to have worked well, although there have been minor troubles due to blocked vents caused by the polymerisation of cyclopentadiene.

(2) *Methanol soda*.[12] In this process the whole crude benzole, or a forerunnings fraction, is treated with methanol (anhydrous synthetic methyl alcohol) and 1.6 lb. of flake caustic soda/gal. of methanol (0.16 kg./l.), by agitation of the reagents with the benzole in the benzole washer. After about 2 hr. agitation, water is added and the residues, which separate out and contain about 80% of the methanol added, are run off, neutralised, and the methanol recovered by fractionation. This process was quite successful and was used on a number of works, but it has now been abandoned at most of them because of the extra trouble involved in recovering the methanol. This operation also has the disadvantage that, unless carefully controlled, it may give rise to objectionable odours. It should also be noted that the benzole, which becomes coloured during treatment, requires a subsequent distillation.

(3) *Ammonium polysulphide (Yorkshire Tar Distillers Ltd.) process*[13] (Fig. 3). This process, which is extensively used in Great Britain for removing carbon disulphide from benzoles, involves stirring the benzole with a reagent consisting of approximately equimolecular proportions of ammonium disulphide, ammonium sulphide and ammonium hydrate. After treatment, which may be given to crude or refined benzoles, or to a forerunnings fraction, the reagent is removed by adding water, and the spent reagent is regenerated by distillation, ammonia being added to restore a strength of 15 g./100 ml. of ammonia. Carbon disulphide is also recovered. One of the attractions of the process undoubtedly has been the arrangement made by the licensers to supply reagent and to take back and regenerate the spent reagent at a number of central points. It was at first feared that some difficulty might arise due to incomplete removal of the reagent, which might cause the benzole to be corrosive because of

hydrogen sulphide. Used properly, the reagent should remove elementary sulphur and, although the process has now been in use for many years, no serious trouble has arisen due to unremoved reagent.

(b) Thiophen removal. Undoubtedly the chief barrier to the production of high yields of motor benzole from crude benzoles has been the necessity for removing sulphur, present in the form of thiophen. In spite of a continuous search for methods of destroying thiophen without the concurrent destruction of unsaturated hydrocarbons, sulphuric acid treatment remains the only practicable method. Because of this, the sulphuric acid treatment necessary for high-sulphur crude benzoles is often so heavy that no advantage can be gained by the use of inhibitors. The benzole is, in fact, stable towards gum formation without further protection. A paper of considerable importance on the sulphuric acid treatment of benzoles was published in 1936.[14] In this paper it was shown that the yield of benzole corresponding, for a particular crude, with a chosen thiophen content in the refined benzole was independent of the quantity and strength of acid used to produce the refined benzole. Thus, provided the refined benzole had the same thiophen content, a small amount of strong acid would produce precisely the same yield as a larger amount of more dilute acid, although there might be other advantages in using the more dilute acid (see ‘ Corrosion of equipment ’ below). Similarly, alterations in the time of reaction, temperature or the addition to the acid of sulphonation catalysts such as vanadium pentoxide, or retarders such as boric or phosphoric acid, had no effect on the yield corresponding with a particular thiophen content.

(c) Other sulphur compounds. One difficulty that has arisen from time to time with the production of inhibited benzoles has been the presence of mercaptans in the refined benzole. These compounds probably do little harm, apart from their objectionable odour, but commercially they cannot be tolerated.

While it is true that some crude benzoles, particularly those recovered from vertical-retort gas, may contain mercaptans, their presence in refined benzoles can generally be traced to one of two causes:[15]

(1) Washing, with sulphuric acid, crude benzole containing hydrogen sulphide.

(2) The addition of an aqueous solution of alkali to the rectifying still in order to prevent corrosion when this still is operating under conditions that may produce hydrogen sulphide, for example when the washed benzole in the still contains elementary sulphur. In this connection a most useful summary of the extensive literature dealing with the reactions of sulphur, hydrogen sulphide and mercaptans with unsaturated hydrocarbons has been published.[16]

Corrosion

(a) Corrosion of equipment. Another difficulty that has increased since the introduction of the inhibitor process has been the production of sulphur dioxide during the final rectification, resulting in excessive corrosion of plant, particularly condensers, and the formation of acid benzoles.

An investigation of this subject showed that increasing the quantity of acid used in the sulphuric acid resulted in an initial rise in the quantity of sulphur dioxide formed, followed by a reduction with higher quantities of acid. Stronger acid (98% by weight) produced a greater maximum quantity of sulphur dioxide than did more dilute acid (brown oil of vitriol, 78% by weight). On the other hand, the stronger acid produced less sulphur dioxide when the quantity of acid was greater.

The unsaturated hydrocarbons chiefly responsible for this production of sulphur dioxide seem to be cyclopentadiene and dicyclopentadiene. There is much to be said, therefore, for the removal of a fore-runnings fraction from the fresh crude benzole, before the dimeric form has been produced by polymerisation. As already stated, once a crude benzole contains dicyclopentadiene, the distillates are liable to contain cyclopentadiene due to depolymerisation. It is not always possible to arrange for the removal of cyclopentadiene from the fresh crude. In these circumstances it has been found beneficial to use the lowest-strength acid that will reduce the thiophen to the required level, and so distil with as much open steam as possible, *i.e.* at the lowest convenient temperature.[17]

(b) Corrosive sulphur. The presence of corrosive sulphur—chiefly elementary sulphur and hydrogen sulphide—in motor fuel is particularly undesirable because of its effect on copper and brass accessories of motor engines. Since the introduction of the inhibitor process and the consequent use of weaker sulphuric acid, corrosive sulphur, when found, has more frequently been in the form of hydrogen sulphide than elementary sulphur. It has been found that the use of the weakest possible acid and a low distillation temperature—conditions which also favour the production of benzoles free from mercaptans—are beneficial in avoiding corrosive sulphur. The use of a final wash of this refined benzole with caustic soda[18] is now becoming standard practice. Usually the benzole is treated continuously by bubbling it through 20 to 40% caustic soda contained in a narrow vertical tank packed with iron rings to disperse the benzole.

Some new facts have come to light during recent years. It has long been known, for example, that corrosion of copper due to elementary sulphur may be profoundly influenced by the presence of naturally occurring corrosion inhibitors. It has now been shown that in benzoles the most likely corrosion inhibitor is cyclopentadiene. This substance may occur in sufficient concentration to have an effect, even in benzoles that have been strongly treated with sulphuric acid.

Another recently discovered fact of some importance is that hydrogen sulphide, even if present in quantities insufficient to cause corrosion, will accelerate the corrosion of copper by elementary sulphur. Thus two benzoles, one containing elementary sulphur and the other hydrogen sulphide, but both present individually in quantities insufficient to cause corrosion, may produce a corrosive benzole if mixed. This fact is of the greatest importance in connection with the storage and blending of benzoles.[19]

Hydrogenation refining

The refining of benzoles by hydrogenation methods is one that is constantly in the minds of benzole refiners. So far there has been little commercial development of this method of treatment, although patents dealing with it are being taken out regularly. The process devised by Yorkshire Tar Distillers Ltd., in which a nickel catalyst and only moderate pressures of hydrogen are used,[20] has in fact been applied to benzole, but apparently the application was not commercially economical.

It appears to be relatively easy to destroy carbon disulphide, which may, however, be removed cheaply by other methods. Conditions sufficiently drastic to destroy the thiophen ring, however, also result in disruption of the aromatic nucleus, with the formation of carbon and deterioration of the catalyst. Immediately prior to the war a hydrogenation process was operating commercially at the Mariendorf Works of the Berlin Gas Co. In this process, iron ore (minette) was used as a catalyst. Benzole was vaporised into coal gas and steam, and fed over the catalyst at a temperature of about 350°C. (660°F.) and at atmospheric pressure.[21] Carbon disulphide was removed, but not thiophen. There is no evidence to show that the process was used in Germany during the war.

Sulphur removal from gas

Although there appears to have been little success in treating benzole by catalytic methods, removal of carbon disulphide from coal gas by such methods is now operating commercially in Great Britain.

The Carpenter-Evans process was one of the first to be operated on a large scale.[22] In this process, reduction of the sulphur compounds in gas to hydrogen sulphide was effected by the use of a nickel catalyst.

The Gas Light & Coke Co. have developed a process,[23] now operating satisfactorily at a number of their works, in which a nickel catalyst, supported on china-clay pellets, is used to oxidise the carbon disulphide and hydrogen sulphide. A controlled amount of oxygen is admitted to the gas which is heated to 160 to 180°C. (320 to 355°F.) before entering the catalyst. The process is one of catalytic combustion and not hydrogenation. Temperature is controlled by adjustment of the oxygen
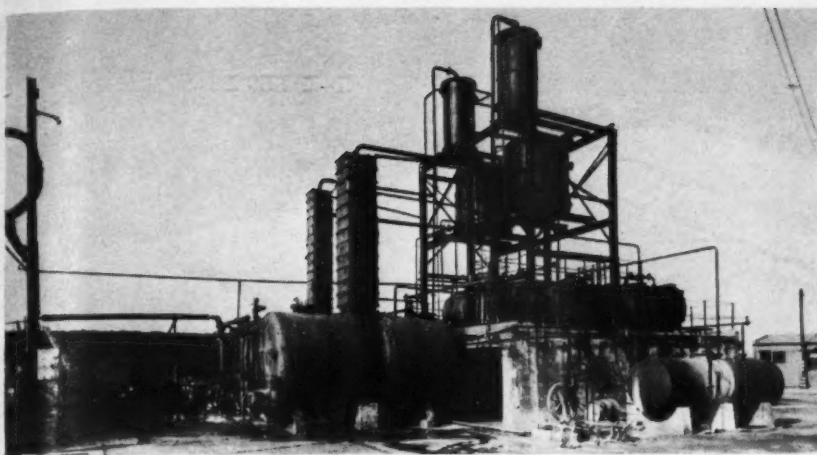
[Photo : Yorkshire Tar Distillers Ltd.

Fig. 3. Plant for the production of ammonium polysulphide reagent for the removal of carbon disulphide from crude benzole. Due to the success of the process, the original plant was soon enlarged to the size illustrated above.

admitted, which is about 0.6 to 0.7% by volume, and there is, in addition to the destruction of the carbon disulphide and carbon oxysulphide, a loss of about 1.3% of the total gas consumed as hydrogen. Nitric oxide and diolefines are also removed.

Another process that has been used successfully is that patented by Maxted and Priestley and erected at the Ravensthorpe works of the Mirfield Gas Co. by W. C. Holmes & Co. Ltd.[24] In this process, a metal thiomolybdate catalyst on a bauxite support is used. The gas is passed over the catalyst at atmospheric pressure and at a temperature of 400°C. (750°F.), the heat being supplied by admitting oxygen, which combines with the hydrogen of the gas. As with the Gas Light & Coke Co.'s process, carbon disulphide is destroyed completely, but only about 10% of the thiophen is attacked.

Future trends

The present outlook with regard to the production of benzole is clouded by the unsettled world conditions, and prophecy must of necessity be speculative. The 1947 figures for the requirements of benzene in the U.S.A. are indicative of the quantities likely to be used in the future for synthetic purposes:

Use				lb. × 10⁶ per annum
Phenol	..	..	..	235
Styrene	..	..	..	280
Nylon	..	..	..	125
Detergents		..	..	50
Maleic anhydride }				
Other uses }			..	400

The position may arise shortly in America when the demand cannot be met without recourse to extraction or synthesis of benzene from petroleum sources. The preparation of benzene in this way, however, cannot compete economically with extraction from coal gas.

The demand for benzene for synthetic purposes in Great Britain can, as yet, be met from high-gravity crude benzole from

coal gas, and there is likely, therefore, for some time, to be a considerable outlet for benzole, particularly low-gravity benzole, as a motor fuel.

For some synthetic purposes, particularly for the preparation of the chlorobenzenes, and for hydrogenation, a high degree of purity is required. Traces of non-aromatic compounds may produce corrosion of distillation plant when chlorobenzenes are distilled, and the unpleasant odour of *Gammexane* (benzene hexachloride) is said to be due to traces of impurity in the original benzole. Sulphur compounds also poison hydrogenation catalysts.

Provided a steady demand for synthesis benzole is assured, the necessary purity can be obtained by conventional refining methods, and so far no better or cheaper methods have been evolved. While the demand for synthesis benzene can be met from high-gravity crude benzoles, it is not likely that recourse will be made to the preparation of pure benzoles by azeotropic distillation from low-gravity sources.

Benzole production up to the present, both in Great Britain and elsewhere, has been mainly in small units and in discontinuous plants. Changes in methods of coal production, such as extensive carbonisation at the pithead, or underground gasification, may eventually lead to more centralisation of refining plant and the greater use of continuous rectification plant.

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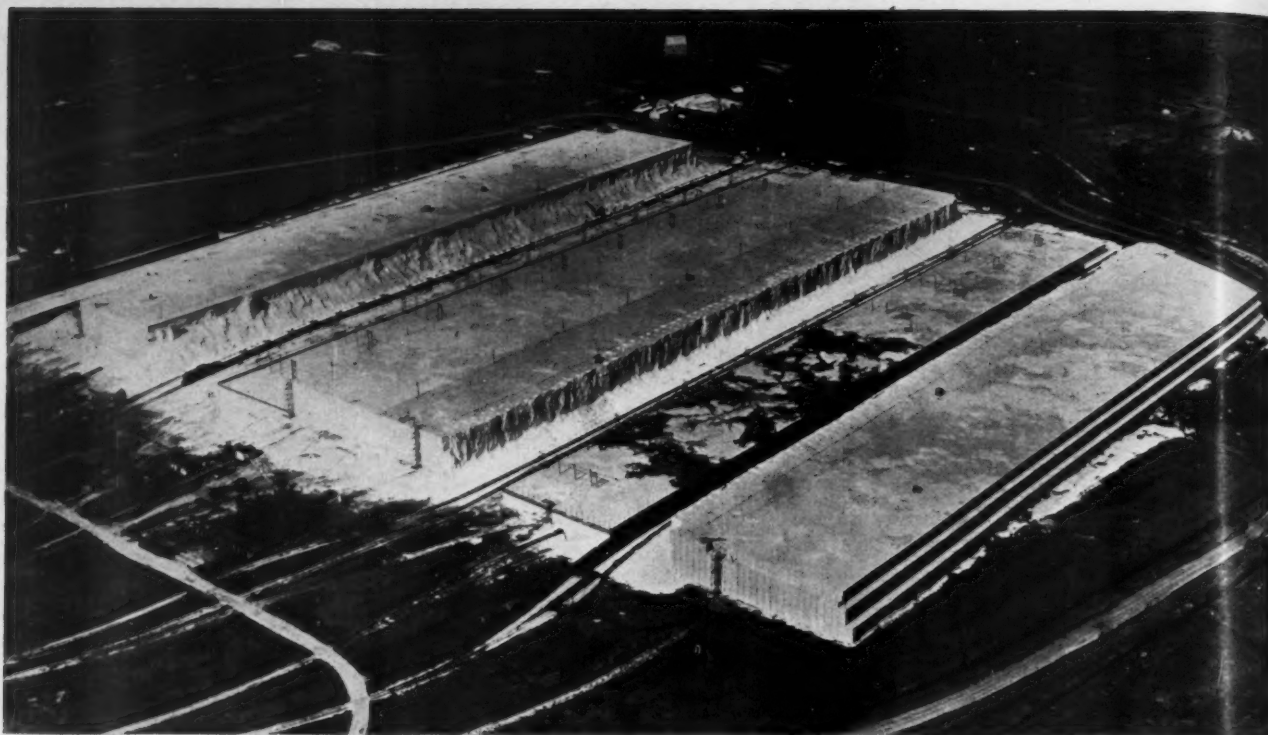
This view of some of the huge sulphur storage vats in Texas, the world's richest source of brimstone, seems to contradict the title of this article. But the Texas Gulf deposits are dwindling fast and no new reserves of any consequence have been found. Hence the U.S. Government's decision to cut sulphur exports which has precipitated a crisis in the many countries dependent upon these supplies for the manufacture of sulphuric acid.

(This and the photograph on page 179 by courtesy of the Shell Magazine)

The Sulphur Famine

The sulphur famine is the main talking point in the chemical industry today. Are there any chances of increasing supplies of mined sulphur and sulphur-bearing ores? What is being done in Britain to produce sulphuric acid from alternative materials like pyrites, anhydrite, spent oxide and petroleum? These questions are answered in the following article.

THE shortage of sulphur continues to be the dominant problem in the chemical industry. There seems little prospect of the Americans restoring their exports of sulphur to the level existing before the cuts were made a few months ago. Since the U.S. produces some 95% of total world output of raw sulphur (last year's output was some 5,000,000 tons), these serious cuts in exports affect chemical industries all over the world. Great Britain, for instance, which last year produced 1,800,000 tons of sulphuric acid, relied upon American sulphur for 60% of this output. Since January 8, owing to the cut in sulphur supplies, total output of acid has been reduced by one-fifth. Although a sulphur allocation committee has been set up in Washington and the British Government is pressing strongly for an increase in supplies, it is unlikely that exports on the former scale will be resumed. Indeed, as is natural, the American chemical industry is becoming increasingly critical of even the present reduced rate of sulphur exports (800,000 tons p.a.) and complains of the

apparent lethargy of the Economic Co-operation Administration in attempts to make Europe less dependent upon U.S. sources of sulphur. This accusation, certainly in the case of Britain, is not completely true, as will be shown later in this article. Before discussing the British effort to grapple with the sulphuric acid crisis, a word on sulphur supplies generally is called for.

Cheap sulphur is scarce

A consideration of the plain facts of the sulphur crisis points to the conclusion that it is not sulphur that is short—what is short is the *cheaply* mined Frasch process sulphur, the product of the American Gulf Coast salt dome industry. The world has other deposits of native sulphur and of low-grade sulphur-containing minerals, but in general these resources cannot be developed at costs that are reasonably comparable with those of the Frasch process. But this situation is not unalterable. So long as the Frasch-mined sulphur dominated world supply there was little encouragement for other sulphur deposits

to be developed or redeveloped. Is it not a little remarkable that the superheated steam extraction of Frasch sulphur, often operated 2,000 ft. below ground level, cannot be imitated in mining surface deposits of sulphur-containing minerals? Must the economic comparison always be between the modern steam-melting and air-pumping Frasch process and the cumbersome hand-labour methods employed in 19th century Sicilian mines?

This question is not merely rhetorical. Already in Columbia a new process for mining surface deposits is being hurried into production (U.S. Pat. 2,537,842). Ores containing no more than 20% of native sulphur are expected to produce sulphur at the current American price for Frasch sulphur. The ores are ground, suspended in water, heated above the melting point of sulphur, cooled, and finally the suspension is subjected to froth flotation. The sulphur is removed with the froth and filtration gives a product entirely suitable for acid manufacture. This process is far more efficient that the crude and

poor-yielding methods traditionally applied to surface deposits. Another similar American process recently reported includes a continuous autoclaving treatment before the flotation operation; this is said to produce 99.5% pure sulphur from low-grade ores at a price that is competitive with the Frasch-mined product. American chemical engineers feel that modern processes of this kind should be applied to some of the sulphur deposits in the rest of the world. It is not an unreasonable expectation when the Frasch-minable reserves of America are so obviously dwindling and when the annual output is insufficient to meet the total demand within the United States.

British sources of sulphuric acid

The development of cheaper methods of extracting mineral sulphur is unfortunately largely out of British hands. Here the only commercially worthwhile sulphur-bearing mineral is anhydrite. This, however, supplies us with only about 30,000 tons p.a. of sulphur. A scheme is afoot to increase these supplies, but the main current effort in Britain is the construction of more plants to use pyrites. At present, imported pyrites supply us with some 100,000 tons p.a. of sulphur. Spent oxide from gas works, now supplying about 100,000 tons p.a. of sulphur is another source likely to be developed. Another prospect is that, with the expansion of home refining, sulphur might be obtained as a by-product of oil. It is estimated that the refineries might eventually supply 70,000 to 100,000 tons of sulphur p.a. Let us consider these sources in more detail.

Anhydrite

At present the only anhydrite-sulphuric acid plant in Britain is that at the Billingham works of Imperial Chemical Industries Ltd. The anhydrite process is an expensive way of producing sulphuric acid. Depending on how much the site has been developed, the plant may cost between £25 to £30 per ton p.a. of capacity, compared with £4 10s. to £6 per ton for a sulphur plant and £12 to £14 per ton for a pyrites plant. However, a by-product of the anhydrite process is cement; for every 1.75 tons of anhydrite processed 1 ton of cement is produced besides 1 ton of acid. This offsets some of the cost, but for the present anhydrite remains an expensive source of sulphuric acid.

In spite of this, it has been decided that the production of sulphuric acid is too vital for an indigenous source like anhydrite to be neglected on account of cost. Accordingly, therefore, plans are afoot to build a plant for the production of 150,000 tons p.a. of acid from anhydrite. The project has been discussed between I.C.I. and a number of important sulphuric acid users and is is likely that it will be financed by the consumers. The most probable site will be Merseyside, where a considerable part of the British chemical industry is located. Anhydrite deposits occur at Barrow-in-Furness and near Carlisle, besides Billingham.

Pyrites

Several acid and fertiliser manufacturers are planning new pyrites plants and are considering the conversion of sulphur-burning plants to pyrites. It is more difficult and costly to switch a plant from sulphur to pyrites than the reverse, but this is being done, notably by I.C.I. Although during the war some 120,000 tons of coal pyrites fines were produced in Britain, the eight concentration plants erected by the Ministry of Supply at various collieries have been closed down, and today most pyrites are imported. Last year, for instance, 205,000 tons were shipped to Great Britain. Main supplier is Spain, where an important group of mines is controlled by a British company, Tharsis Sulphur & Copper Co. of Glasgow. This company exported 400,000 tons of pyrites last year and expects to increase shipments to the U.K. during the next eighteen months. Spain's total exports of pyrites are expected to amount to 1,300,000 tons this year. Labour and power problems must be overcome if production is to be greatly increased.

As mentioned, pyrites plants are more complicated and costly than the straightforward sulphur combustion plants. A plant for producing, for instance, 10,000 tons p.a. of acid from pyrites costs £250,000. However, the Government is said to be giving top priority to the erection of pyrites plants and to the conversion of sulphur plants.

Synthetic Chemicals Ltd. of Knottingly, Yorks., are shortly to bring into operation a pyrites plant. Courtaulds, the rayon manufacturers, are also reported to be building a pyrites plant, while recently the West Norfolk Farmers' Manure & Chemical Co-operative Co. Ltd. of King's Lynn announced plans for a pyrites plant to employ Freeman Nichols flash roasters.

Spent oxide

This is a by-product of the gas industry and is the main indigenous source of sulphur. Gas works have sometimes found it difficult to produce oxide of uniform quality or containing sufficient sulphur for the chemical industry and, in any case, the value of spent oxide is further limited, as it can only be used economically in connection with chamber acid plants. However, there is reason to believe that modifications in the practice of gas purification may result in the production of material that not only is more uniform but also has a higher sulphur content. It is also hoped to reduce both the capital cost of the plant and to produce higher-strength acid. Other lines of investigation are concerned with the extraction of sulphur from spent oxide and methods of gas purification which give elementary sulphur as an end product. Production from spent oxide, however, cannot be increased much further as present and planned plant capacity can absorb all available spent oxide.

A new acid plant utilising spent oxide is to be brought into operation by Lawes Chemical Co. Ltd.

Sulphur from petroleum

Of the other sources of sulphur available to British industry, the petroleum industry seems the most promising; as mentioned, it has a potential of up to 100,000 tons p.a. H. Heukensfeldt-Jansen, writing in the March 1951 issue of *Shell Magazine*, states that the manufacture of sulphur from the refinery gases liberated during topping and cracking operations necessitates the provision of equipment additional to that required for ordinary refinery operations. This equipment comprises two main units, the first of which extracts H_2S from refinery gases and the second of which converts the H_2S into sulphur, which may be made available as such or converted into sulphuric acid.

Zinc blende and coke oven gas

The shortage of zinc makes discussion of zinc blende as a substantial source of sulphur rather academic in the present context. However, it may be recorded that the manufacture of zinc from zinc sulphide concentrates which are roasted produces a gas suitable for H_2SO_4 manufacture.

To recover the sulphur present in coke oven gas at present going to waste would involve the erection of expensive plant and the resultant sulphur would, in any event, not replace more than 7 to 8% of total British requirements.

Summing up

To sum up British efforts to overcome the sulphur shortage, it seems that the main developments are concerned with pyrites plants, with the scheme for a new anhydrite plant coming a close second. Spent oxide is another substantial source, but further expansion does not seem immediately possible. The oil industry is a potentially large source of sulphur. Zinc blende and coke oven gas are unlikely to make big contributions.

Most of these efforts are of a long-term nature; they do not provide an immediate replacement for the one-third cut in supplies of American sulphur. Bearing in mind that imports are more likely to decrease than to increase, Britain will be lucky to maintain sulphuric acid production at even the present reduced level. However, if the present schemes are carried through vigorously and quickly, the situation may begin to improve within eighteen months or so. Looking ahead five to ten years, Mr. W. A. M. Edwards, Commercial Director of the General Chemicals Division of I.C.I., foresees that there will be very little American sulphur indeed for acid manufacture in Britain. To meet this eventuality, he thinks it will be necessary to convert existing plants and to build new plants to burn pyrites and anhydrite to the extent of at least 1,000,000 tons annual capacity.

Methods of Sulphur Recovery

In view of the present shortage of sulphur, caused largely by the reduction of U.S. exports to Europe, it was thought that there would be renewed interest in the various methods adopted in Germany to combat a similar shortage lasting over a long period. In this connection the captured German documents and microfilmed reports, as well as the well-known reports by Allied investigators, should assume a new and special significance. All this material is held by the Technical Information and Documents Unit of the D.S.I.R. for the benefit of British industry. Several enquiries have been received recently on the sulphur problem, and it was felt that an effort should be made to bring this material to the notice of industrial firms and other organisations likely to be affected by the present shortage. It was decided that this should take the form of a short bibliographical survey, making the list of references as complete as possible, but leaving the plums to be picked out by those interested in the problem. The opportunity has been taken to include in the report some references to information, obtained recently, on natural sulphur supplies outside the United States.

Recovery of sulphur in Germany

THE German Government had recognised, long before the 1939-45 war, the necessity of economising in sulphur, in view of the fact that they were dependent mainly on supplies from overseas. As a result, they set up an organisation controlling production, imports and distribution of sulphur. This organisation, known as Schwefel G.m.b.H., Berlin, was responsible for collecting data from all concerned relating to sulphur and sulphur recovery. It instructed individual firms regarding the best methods to be adopted in preserving sulphur supplies, and it was the negotiating body with foreign firms on sulphur imports. From documents emanating from this organisation we know that, for example, in the year 1943-44 a total of approximately 90,000 tons of sulphur was recovered from sulphur-containing wastes, including industrial gases, in the various industries.

In addition, economies were made through the setting up of plants for the manufacture of sulphuric acid from anhydrite. Through this project alone, 20,000 tons of sulphur was saved in 1943.

Regarding the recovery of sulphur from coal, it may be noted here that the Germans had recovered 70% of the available sulphur in coal in the Ruhr by the end of 1944 (FD 5558/47).

Sulphur recovery processes

Probably the most important process in Germany for the recovery of sulphur was the *Alkazid* process, coupled with the catalytic oxidation of hydrogen sulphide in Claus ovens. Although there is considerable information on other processes, the most detailed information available relates to these processes.

It will be convenient to divide the various methods adopted into (*a*) dry recovery processes, (*b*) wet recovery processes and (*c*) processes in which an organic liquid is used as the scrubbing medium.

(a) **Dry recovery processes.** The use of iron oxide is probably the oldest known process of sulphur recovery. The iron oxide reacts with hydrogen sulphide and, therefore, the presence of another substance increasing the alkalinity of the medium is of advantage. Thus traces of ammonia have been used to increase the rate of absorption of hydrogen sulphide (FDX 487, frames 4500-4503; FDX 503, frames 6574-6591; FDX 236, frames 858-859 and FD 4845/47).

Where the concentration of hydrogen sulphide in the gas is low (under 5 g./m.^{3}), *e.g.* in producer gas or water gas, active carbon (*Carbonorit*) is often used in place of iron oxide. The absorption over *Carbonorit* was followed by scrubbing with ammonium sulphide or CS_2 (FD 1345/49 and TOM Reel 207, III, Part 1, frames 85-87).

The principles of dry recovery processes, especially as applied to coke oven gases, are described in *Gas und Wasserfach*, 1938, **81**, pp. 822-828.

(b) **Wet recovery processes.** In these processes the oxygen carrier is present either in solution or in colloidal suspension.

The *Thylox* process, well known in America, uses a salt of thio-arsenic acid. A description of a plant making 7,500 kg./day of sulphur by this process is given in a microfilm (FDX 642, frames 8234-8240; also *Chem. Fabrik*, 1939, **12**, p. 15).

In the potash process the scrubbing medium is potassium hydroxide, which is subsequently warmed to release hydrogen sulphide (TOM Reel 207, IV, Parts 2 and 4, frames 92-93, 99-100).

A solution of complex cyanides is employed in the Staatsminjen-Otto process. A short description and references to published literature on this process are given in FDX 709, frame 38355 (*cf.* also FDX 236, frames 858-866).

The *Katasulf* process developed by the I.G. Farben is based upon the oxidation of hydrogen sulphide gas to SO_2, which is absorbed by ammonia. It is specially suited to coke oven gases where ammonia is available on the site. This process is described in a publication (not held by T.I.D.U.), *Chem. Fabrik*, 1938, **11**, p. 10 (*cf.* also FDX 236, frames 858-866, and FDX 89, frames 485-486).

(c) **Processes using an organic scrubbing agent.** The two most important processes in this field are the *Girbotol* and the *Alkazid*. Both are essentially ' enriching ' processes, in that the hydrogen-sulphide-containing gases are led through organic absorbing agents, and subsequently a gas rich in hydrogen sulphide is released. Thus they provide the first stage in sulphur recovery proper. The hydrogen sulphide is then oxidised, either to sulphur in Claus ovens, or directly to sulphuric acid in the *Lurgi* process (FD 5558/47 and FD 2250/48).

The *Girbotol* process uses mono-, di- or tri-ethanolamine, or diamino propanol (FDX 647, frames 2962-2963, and FDX 89, frames 479-489).

The *Alkazid* process was claimed to be more efficient than the *Girbotol* process. The chemical compound used in aqueous solution consisted of the alkali salt of amino propionic acid. The Germans subsequently developed quite a number of compounds for this purpose, some of chemical constitution related to this original compound, others entirely unrelated. As will be seen from the bibliography, information held by T.I.D.U. includes instructions concerning the methods of preparation of chemicals, the corrosive nature of the *Alkazid* lyes, plant drawings, flow sheets, etc.

Acid from anhydrite

The largest plant for this purpose was installed at Wolfen, which is now in the Russian zone. The basis of the process was the reduction of calcium sulphate with coke; the by-product is a Portland cement clinker. Experiments have also been carried out in connection with the production of *sulphur* by the reduction of calcium sulphate (anhydrite), without, at the same time, acquiring valuable by-products such as cement. These experiments did not, however, result in the erection of large-scale plants, as the method was not considered economic (FD 5034/47).

Sources of sulphur outside the U.S.

The main natural sulphur resources outside the U.S. are those in Sicily, where

over 380,000 tons were produced in 1938 and, although there was a slump in the production during and immediately after the war, it is expected that production of raw fused sulphur in Italy will reach a level of about 200,000 tons in 1951. Two reports containing up-to-date information on the Italian sulphur industry have been received recently from the Commercial Department of the British Embassy in Rome, and are held at T.I.D.U. under the references B/TP 200/1944 and B/TP 200/1945.

There is also natural sulphur in Japan; statistical information on sulphur production in Japan, including information on exports during 1928 and 1936, is available in a document, reference FD 45/51.

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(a) Statistical

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(b) Reviews

FDX 89:
Frames 452-67. Patent review and discussion of methods of removal of H_2S and CS_2 from waste gases, with special reference to sulphur recovery in viscose production (Vereinigte Glanzstoff Fabriken, Obernburg and Seehof).
Frames 479-489. Review of sulphur recovery from waste gases, giving details of Katasulf, Alkazid and Thylox processes from the point of view of viscose production (Vereinigte Glanzstoff Fabriken, Obernburg and Seehof).
Frames 425-429. The conversion of H_2S and CS_2 in waste gases from viscose rayon production to H_2SO_4 by combustion and oxidation to SO_2 by a wet catalytic method.

FDX 236, frames 858-859. Review of sulphur-recovery methods (Phrix-Werke A.G., Hamburg-Altona).
FDX 709, frames 38352-38360. An article from *Das Gas und Wasserfach* on modern desulphurisation processes.
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(c) Sulphur from coal

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(d) Potash process

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Part 2, frames 92-93. Removal of hydrogen sulphide by the potash process. Drawing and layout.
Part 4, frames 99-100. Description of regeneration of potash reagent, with diagram.
Part 5, frames 101-104. Removal of hydrogen sulphide by Koppers potash process.
Part 7, frames 111-126. New process of wet desulphurisation of coke oven gases.

(e) Absorption by active carbon

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(3) Thylox process

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Item 6. Flow sheet showing an NaOH wash, following the Alkazid absorbtion.
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FDX 595, frames 1811-1823. Determination of formic acid in Alkazid solutions (I.G. Farbenindustrie A.G., Leuna).
FDX 612, frames 41-104. Preparation of Alkazid (I.G. Farbenindustrie A.G., Ludwigshafen).
FDX 780, frames 7075-7083. Recovery of carbon dioxide from Alkazid waste gas. A method of separating carbon dioxide and sulphuretted hydrogen is given (Chemische Werke Hüls in Marl, Westphalia).
FDX 797, frames 978-981. Alkazid process in viscose rayon production (Vereinigte Glanzstoff Fabriken, Obernburg).

(6) Claus ovens

BIOS Final Report 1702. ' The Manufacture of CS_2 with Notes on Sulphur Recovery.'
FD 2700/46. Reports on the working of Alkazid-Claus plant (I.G. Farbenindustrie A.G., Ludwigshafen).
FD 3376/46. Erection and efficiency of Alkazid installation, coupled with Claus ovens (Ammoniakwerk Merseburg G.m.b.H., Leuna).
FD 3384/46. Patent applications relating to Claus ovens (I.G. Farbenindustrie A.G., Ludwigshafen).
FD 327/47. Recovery of sulphur from H_2S by the Claus process (I.G. Farbenindustrie A.G., Premnitz).
FD 871/47. Chemical equilibrium in the Claus process (Ammoniakwerk Merseburg G.m.b.H., Leuna).
FD 717/48. Drawing of Claus oven installation at I.G. Farbenindustrie A.G., Leverkusen.
FD 2508/48. Details of equipment and some technical data relating to Claus plant (A.G. Saechsische Werke and Braunkohle-Benzin A.G., Boehlen).
FD 3134/48. Details of Claus oven (see also TOM Reel 195, part 8) (Ammoniakwerke Merseburg G.m.b.H., Leuna).
FD 3289/48 (FIAT Final Report No. 1015). ' Oxidation of Hydrogen Sulphide to Sulphur in Claus Ovens.'
FD 1469/49. Constructional drawing and details of a multicylcone for sulphur recovery (Lurgi Apparatebau-gesellschaft m.b.H., Frankfurt).
FD 1156/50. Improvement in Claus ovens and description for the operation of Claus processes (I.G. Farbenindustrie A.G., Ludwigshafen).
FDX 693, frames 7749-7754. Draft patent application by Ruhrchemie A.G., relating to burning of hydrogen sulphide to sulphur.
FDX 709, frames 38392-38395. Patent application concerning the oxidation of hydrogen sulphide into sulphur dioxide (Anorgana G.m.b.H., Gendorf).
TOM Reel 132:
Items 7-10, 13-16 and 18-19. Flow sheets and operating instructions relating to the Claus process.
Item 27. Patent announcement dated June 27, 1939, relating to catalytic recovery of sulphur from gases containing H_2S.
Item 30. Drawing and layout of Claus unit.

(7) Sulphur and sulphuric acid from anhydrite

CIOS Report No. XXIV-22. ' The Electrochemical Industry Bitterfeld Area.'
BIOS Final Report No. 678. ' Manufacture of Cement and Sulphuric Acid from Anhydrite (I.G. Farbenfabrik, Wolfen).'
FD 2767/46. Production of and plant for Portland cement and sulphur by the Lurgi method (Wirtschaftliche Forschungsgesellschaft m.b.H., Niedersachswerfen).
FD 5034/47. Experiments on the production of sulphur from calcium sulphate (Metallgesellschaft A.G., Frankfurt).
FD 3072/48 (BIOS Report No. 830). Production of sulphuric acid, sulphur and phosphatic fertilisers.
FD 1366/49. Working up of anhydrite for the production of sulphuric acid and cement (Lurgi Gesellschaft für Chemie und Huettenwesen m.b.H., Frankfurt).
FDX 164, frames 6261-6268. Production of sulphur from gypsum (I.G. Farbenindustrie, Ludwigshafen).
FDX 597, frames 1421-1433. Sulphur-from-gypsum experiments (I.G. Farbenindustrie, Leuna).

Drying Technique

By R. R. Clegg, A.M.I.Mech.E., A.M.I.H.V.E.

(Tomlinsons [Rochdale] Ltd.)

USUALLY it is the physical form of the wet material which determines the way in which it must be handled in the drying machine and the physical characteristics which determine the operative conditions during that process, so that, while the mechanical arrangement of the machine must accommodate the former, the aerodynamical and thermal design must satisfy the latter. In the drying process the temperature of the stock must be raised to such a degree and in such a manner that the excess moisture in the stock will be evaporated and the vapour continually removed from the system. This means, firstly, that heat has to be transferred from the heating medium to the stock and, secondly, that the atmosphere must be thoroughly ventilated.

There are four main types of driers: (1) contact driers; (2) convection driers; (3) vacuum driers; and (4) radiant-heat driers.

Contact driers

Flat and cylindrical drying surfaces. Contact driers take the form of flat or cylindrical contact surfaces, according to the characteristics of the stock. With the flat type the stock is usually stationary within the machine throughout the drying cycle, or is stationary while the platen surface is in contact with it. With most driers, however, the contact surface is cylindrical and in motion.

The conditions existing on a contact surface can be considered analogous to those existing in any other form of thermal drying machine and thus may be divided into three stages: (1) The phase during which heat is given to the stock and the moisture which it contains, raising their common temperature with little or no evaporation; (2) the phase during which the bulk of the moisture is evaporated; and (3) the phase during which the final moisture is evaporated, the rate of evaporation progressively diminishing and the temperature of the stock progressively increasing.

The characteristic feature of the contact drier is that the temperature of the contact surface is above that of the ambient air and usually above the atmospheric boiling-point of water. Heat transfer between stock and air will be always in the direction from stock to air (and not, as in the case of the convection drier, from air to stock), so that the wet-bulb temperature of the surrounding atmosphere will have no direct relationship with the temperature of the stock.

Heat for vaporising the moisture enters the stock from that side which is in contact

In this article, which is a summary of a paper given before the Institute of Fuel as part of their symposium, 'A Study of Drying,' the author surveys the main types of drying machines in current use, with particular reference to the design characteristics demanded by a wide range of materials to be dried. It may be read in conjunction with the articles on drying which appeared in the February issue of INTERNATIONAL CHEMICAL ENGINEERING.

with the heated surface, and the vapour evolved either passes through the wet stock or emerges from the plane of contact (the latter is, however, unlikely because of the pressure exerted by the stock on the contact surface).

Efforts must be made to induce turbulence of the heating mass close to the shell. The shell must have a high thermal conductivity and its surface must be clean, smooth and perfectly cylindrical.

An air stream directed on to the outer surface of the stock will help to increase the overall rate of heat transfer. The author had always considered that such an air stream would not be effective unless its temperature exceeded that of the stock surface, but recent experiments by the British Cotton Industry Research Association have shown that such may not be the case.

There are four alternatives to the use of steam as the heating medium, namely high-pressure hot water, heated oil, electrical elements and direct firing. Steam has the advantage that it is more easily applied, is often available in large quantities at low cost as pass-out steam and that, compared with hot water or hot oil, a smaller weight is required for a specific drying duty. On the other hand, the heat transfer coefficient from water or oil to the solid shell is greater than that of the vapour to the shell, but this advantage is offset by the need to employ circulating pumps when using hot liquors. When steam is used it must be dry, saturated, and free from air if the potential heating capacity, as indicated by the pressure gauge, is to be realised.

Film or roller driers. These machines, which are used extensively for the drying of liquids, pastes or slurries, consist of a drum on the heated surface of which the wet stock is spread in a uniform layer or film. The drum is rotated at such a speed that the drying cycle is completed in less than one complete revolution, so that a continuously available drying surface is presented at the point of feed. Dependent upon whether the stock is to be completely or partially dried, the surface will be either flat or smooth or grooved and finned. The machine may be built either with a single drum or with a number of drums.

The dry stock is removed by scraper knives or by individual doctor blades, and leaves either in the form of flakes or granules (smooth drum surface) or partially dried sticks (grooved drum).

Convection driers

These are drying machines in which a stream of moving gas (usually air) is utilised as the principal heating medium. They constitute the largest and most varied single group of drying machines, and are able to handle practically every type of stock. They therefore vary greatly in size, design and operating procedure, but all include air-circulating equipment, air-heating equipment and a stock-supporting mechanism, each of these items being suitably protected against loss of heat by radiation.

The essentials in the design of a convection drying machine are: (1) To ensure uniformity of air flow in relation to the stock; (2) to ensure the maximum rate of heat transfer, by providing as high a rate of air flow across the surface as is compatible with its physical characteristics and the operating point within the drying cycle; and (3) to ensure the optimum relationship between the temperatures registered by the wet- and dry-bulb thermometers. In practice, it is found that a system of vigorous air recirculation will satisfy the second and third requirements, but a compromise has often to be accepted with respect to the first, since the degree of uniformity in the contact between air and stock is dependent upon the shape, size and formation of the wet material. In the attempt to make this contact more intimate and uniform many different types of convection driers have been evolved.

The drying room. The most elementary form of convection drier consists of an enclosed space in which the stock is either hung or stacked and through which air is allowed to flow under the influence of natural convection. As this arrangement satisfies none of the requirements mentioned above, slow and uneven evaporation may be expected; it is found that pockets of stagnant air and vapour begin to form near which the rate of evaporation is negligible.

Batch driers. This is probably the most popular design of convection drying machine in use today, chiefly because of its low initial cost and the ease with which it can be made to handle materials of widely differing characteristics. It is a direct development from the drying room, but, unlike that arrangement, satisfies the three requirements.

Batch drying machines may be either single- or multi-chamber, and each chamber is fully loaded with a charge of wet material, which remains until it is dry.

The air circulation within the compartment may be either horizontal or vertical, according to the nature of the stock, the usual direction being horizontal.

The decision to employ upward or downward air flow is sometimes taken on the erroneous assumption that (a) the air will move upwards through the chamber by natural convection and (b) the moisture within the stock will gravitate towards the lower extremities. Quite apart from the fact that natural-convection effects are of little consequence in forced convection, the heating elements may be so located in relation to the wet stock and to the point of heat input that no advantage is gained from the effects of natural convection.

Further, before the stock is placed in the drier, its moisture content should be reduced below the point of fibre saturation. In the few cases where this is not practicable, an intermittent reversal of the air-flow direction can be of advantage. With light, threadlike stock, which must of necessity be suspended within the drying compartment, downward flow will produce less entanglement of the filaments than upward.

No decision should be taken regarding the method of supporting the trays until due consideration has been given to the conditions governing the whole cycle of production. The primary aim should be to reduce to a minimum the manual effort required to operate the plant.

Tunnel driers. For high rates of output the multi-chamber batch drier is uneconomical to operate and, instead, a tunnel drier is used. This is a development from the batch drier, the drying chambers being placed end to end in the form of a continuous compartment through which trucks carrying the stock supported on trays, rods or in clips are mechanically propelled at predetermined intervals.

Multi-band conveyors. A further development is the continuous-band conveyor-type tunnel kiln. This consists of an extended drying compartment or tunnel through which pass one or more conveyors carrying the stock. This enters the tunnel by the uppermost conveyor and falls successively from conveyor to conveyor as it progresses through the machine, until it is finally discharged from the bottom conveyor.

Because of the resistance to air flow created by the layers of stock within such a multi-conveyor machine, it is not gener-

ally possible to employ zonal recirculation for drying loose stock. In such cases it is an advantage to combine a section of single-conveyor system with a section of multi-conveyor, the two forming a single unit. This type of drying unit can be applied with advantage to materials which have a constant-rate period short in comparison with the falling-rate period, especially if floor space is at a premium and it is possible to accommodate a machine of considerable height. By this means one can secure the advantage of the single-conveyor machine during the constant-rate period, whilst the less stringent requirements of the falling-rate period are more easily met by the multi-conveyor section.

Single-band conveyors. Single-conveyor drying machines have the advantage that they give positive control of temperature, humidity, drying time and rate of intake of the stock. A simple redisposition of the air-circulating equipment will permit of upward or downward flow, or of any combination of these, along the length of the machine.

The hot air can either be admitted to the drying chamber freely or it can be kept under control by means of impact jets until contact is made with the stock surface. The choice between these two methods depends upon the stock characteristics; in general, impact jets are used for stock which can pass continuously through the chamber in the form of a layer or piece and is not subject to excessive case-hardening conditions. Impact jets may be less useful if the stock is irregular in form or is held in suspension, in which case it is difficult to maintain the control of the air stream over the whole of the stock surface.

Design considerations. When the stock is of loose formation the band is so constructed as to present a continuous surface from the point of feed to the point of discharge. It may be fabricated from a belt of woven mesh or from a series of individual cross-bands. These latter, depending upon the fineness of the stock, are either of woven-mesh or of perforated-plate construction and are firmly secured to the conveyor chains running the length of the tunnel.

Side guards of the moving and of the stationary types are secured to the conveyor bands and to the tunnel respectively, to prevent seepage of material on to the chains and on to the floor of the tunnel.

When the stock is held in suspension the continuous conveyor band is replaced by individual cross-members, either detachable or forming an integral part of the band. The shape of the material and the method of suspension decide the pattern to be adopted.

Except in those machines where a straight-through air flow is essential, an attempt is made to pass the air stream more than once through the stock on the conveyor band. The extent to which this principle can be applied is determined by the permissible capital outlay on the

equipment. It finds its most extended application in the complete zonal recirculating system. In nearly every case the nominal direction of flow of the warm air is vertical—either upward or downward, according to the stock characteristics of the stock. By adopting the primary and secondary system of air circulation it is possible to provide the conditions of high temperature and velocity permissible during the constant-rate period and, by a simple redisposition of fan and heating elements, to satisfy also the conditions required by the falling-rate period.

The continuous-type tray drier. Two of the principal disadvantages associated with the operation of the tray- or shelf-type drying machine are: (a) It is extremely laborious and costly to load and unload the individual trays; and (b) it is difficult to ensure absolute uniformity of drying conditions from tier to tier and across the projected area of the trays.

To overcome these inherent disadvantages the continuous-type tray drier was evolved. This machine consists of a central circular tower carrying shelves arranged in a tier and so constructed that the material to be dried can fall from shelf to shelf either directly or by a spiral path. The tower may either be made to rotate *en bloc* against stationary scraper bars or, alternatively, may remain stationary while the scraper bars rotate across the tray surfaces.

The wet material is charged into the tower at the top and the dried product is discharged at the bottom; air is circulated between the shelves as in the simple type of tray drier.

The advantages of this machine are those to be obtained from interzonal and progressive recirculation. It is possible to arrange for the stock to be in contact with low-temperature dry air at the point of material discharge and with high-temperature moist air at the point of material entry. Further, the continual agitation of the material on the trays as it passes through the machine constantly presents a new wetted surface to the air stream and thus increases the rate of evaporation.

The disadvantage associated with the machine is the fact that the material must, of necessity, cascade freely from tier to tier and in so doing must pass through the stream of moving air crossing the tray surface, so that, unless the velocity of the air stream is extremely low or the material reasonably heavy, a considerable amount of the material to be dried is entrained by the air and is deposited in the various crevices and compartments of the machine.

Spray driers. Spray driers are designed specifically to handle liquid stock, to dry the stock by convection and to produce a dried product in finely powdered form.

Essentially, the spray drier consists of a mechanism for atomising the liquid stock and bringing the droplets thus produced into intimate contact with a stream of hot air while making provision to dispose of the dry material and to remove any

which may have been entrained by the stream of hot air.

The conditions in this machine are almost ideal for convection drying. The rate of drying will depend on, amongst other things, the size of the globules, which is determined by the atomiser. There are three principal methods by which atomisation is achieved: (1) The liquid can be sprayed at very high pressure through a fine orifice of an atomising nozzle; (2) a fine jet of the liquid is directed against the upper face of a disc or wheel which is revolving on a vertical axis; and (3) by using compressed air with a number of nozzles. As the globules leave the atomiser they quickly lose their kinetic energy and fall easily through the drying chamber, through which is also flowing a stream of hot air or flue gas.

While the majority of the stock may be deposited in the lower regions of the drying compartment, some of the stock will become entrained in the gaseous stream and will go forward to the point of discharge. Precautions have to be taken against the loss of the product to atmosphere, and filtration equipment must be installed.

Pneumatic driers. These are similar to the spray drier in that the wet stock is entrained in the stream of hot gas passing vertically through the drying compartment.

By adjusting the velocity of the gases according to the characteristics of the stock to be handled, it is possible for the moving stream to select the dry material and carry it forward while rejecting the wet material. The latter is either maintained in a state of semi-turbulence within the heating tube until it has been sufficiently dried or else is by-passed to the feeding device and recirculated.

The gaseous products of combustion are used as the heating medium and are circulated at a high velocity through a tubular form of drying compartment which may be over 30 ft. in height. By thus bringing the wet material into immediate contact with the high-temperature heating medium a high rate of evaporation is obtained and, for this reason, the machine is sometimes called a ' flash drier.'

An essential feature of this system is that the particles of wet material as admitted to the drying compartment should be approximately similar in size, weight and moisture content, otherwise scorching may occur.

Rotary driers. The rotary-type continuous drying machine is especially useful for drying materials which are able to withstand the impacts and abrasion caused by continual and vigorous cascading and which do not deteriorate materially when exposed to high-temperature conditions. Essentially it consists of a horizontal tubular shell through which the stock and (perhaps) the gaseous heating stream are passed.

Depending upon the limiting temperature conditions, the hot gases are forced directly and initially through the central

core of the shell or are first passed through jacketed cavities on the outside of the shell. Alternatively, the gases may be retained completely on the outside of the shell, which is then built within what is virtually a combustion chamber.

As with the tunnel drier, the hot gases may flow either in the same or the opposite direction to the stock and, with certain specific designs, a degree of interzonal circulation is possible. The shell may be made to rotate or, alternatively, it may remain stationary; in the latter case, an independent central heating element revolves alternately through the stock and through the heating medium.

If the air stream passes through the centre of the shell the machine is essentially a convection drier, although conduction from the shell and from the flights, together with a degree of internal radiation, assists in the overall heat transfer.

In those machines in which the heating medium is retained on the outer surface of the shell, the drying process depends upon conduction assisted by radiation.

In the static shell-type rotary drier the heat transfer is mainly by conduction. The material to be dried remains almost static during transit through the drum, being retained within the lower half of the latter, so that the concentric heating elements of the rotor are partially submerged in the stock and partly exposed to the stream of hot gas flowing across the surface of the stock.

Vacuum driers

Nearly all vacuum driers are essentially contact-drying machines. The whole of the operation of imparting heat to the stock is carried out within a hermetically-sealed space in which a high vacuum is maintained. The result is that there is a very considerable difference in vapour pressure between that of the liquor to be evaporated and that in the ambient space, even though the temperature difference between the stock and the liquor is relatively low.

Vacuum driers may be of the shelf type, the continuous conveyor type, the rotary type or the drum type. They are widely used in the chemical industry and for certain food-preparation processes. The maintenance work needed to preserve a high vacuum is the main disadvantage of this type of drier. Another disadvantage, which applies only to the shelf-type machine, is the labour and the loss of effective drying time involved in the operations of loading and unloading.

Radiant-heat driers

The principles involved in radiant-heat driers differ fundamentally from those of contact (conduction) and convection driers. The transfer of heat energy depends entirely upon radiation and absorption, i.e. radiation by the heat source and absorption by the stock.

It follows that only those bodies which possess high properties of radiation when hot should be used as emitters, while those which are poor receivers are not the best types of stock for such treatment.

For the stoving of lacquered surfaces on metal-base components, the ' infra-red' system has proved very effective. A wavelength of the radiant energy is such as to pass through the veneer of lacquer with little or no effect, the heat being absorbed by the metal base. The temperature of this base rises steeply and thus the lacquer is dried from the inner side by conduction.

On occasions it is an advantage to assist the drying potential of the radiant heat by a suitably applied convection current, for which purposes the waste products from the direct-fired element can often be employed.

New standards

Fire buckets. B.S. 1689 has the object of encouraging a reduction in the number of sizes and designs of fire buckets to provide greater uniformity. The sizes specified have been selected so that the buckets can be easily handled by women. The standard covers both the round-bottom and flat-bottom type of bucket and gives details of the construction and general dimensions, together with drawings showing the shape of the finished buckets. Copies of this standard cost 2s., post free, from the British Standards Institution, 24 Victoria Street, London, S.W.1.

Coal Tar pitches for building purposes (B.S. 1310: 1950) now includes, in addition to the requirements for the five normal high-temperature pitches for building purposes previously covered, two special types of pitch for use in pitch mastic flooring, i.e. low-temperature pitch and steam-blown high-temperature pitch, the special feature of these pitches being that they can be used at temperatures up to 160°C. without emitting obnoxious fumes.

The origin and general characteristics of the pitches are laid down and methods of test are included for softening point, specific gravity, matter insoluble in toluole, ash, water content and distillate. These tests are based upon methods given by the Standardisation of Tar Products Test Committee in ' Standard Methods for Testing Tar and its Products,' 1950 edition, modified where necessary. In addition, the special pitches are required to pass a fumes test. Examples of the use of the various pitches for building purposes are given. Copies of the standard cost 2s., post free, from the Institution as above.

New Plant and Equipment

Bin discharger for fine powders

Discharging flour and other finely ground materials from bulk storage can be fraught with danger as well as difficulty. They will not flow and find their own level like liquids or more granular substances, and if an aperture is uncovered at the bottom of a container only the material which is directly above the hole will run out, and a long empty shaft will be formed vertically through the bulk. In a silo or other bulk container with a capacity of several tons, the sudden shifting or avalanching of the material upon such an air space could cause a severe and damaging compression akin to an explosion.

Other means working at the bottom of a bin can produce the same results, and hoppering the bin to give a funnelling effect increases this tendency. Some form of mechanical extractor which will remove the material evenly from across the whole floor of the container is needed.

The mechanical problems set by such a device are claimed to have been solved in the new Redler bin discharger made and marketed by the Bulkbin Co. There are 3-in. transverse slots at intervals across the width of the bin floor; specially designed metal 'knives,' attached at each end to driving chains, move slowly across this floor and in one action slice off and push the bottom layer of the material along so that it falls through the slots. But to move metal bars steadily and continuously under the weight of a full bin load would put enormous strain on heavy chains needed for such a purpose. This new system, however, enables the discharger to be driven by a very low-powered motor. It overcomes the difficulty by moving the special knives one end at a time; the chains take up the drive alternately, and each knife moves with a diagonal, cheese-cutting action on the principle of a lever.

The driving chains are operated by a spur gear, and the start of the movement of the chains takes place at the moment of maximum leverage, so that the force applied to start the metal knives moving is very great. This action is such that the discharger will start immediately, no matter how long the material has been allowed to stand and 'set.'
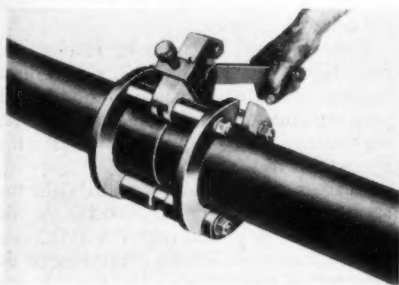
Pipe alignment clamp.

The slotted bottom of the container is set at an angle so that the slicing action is downhill; the chains with their metal knives return along a level false bottom and sweep out the fallen material in a quiet and continuous discharge, the rate of which is variable at will.

The discharger allows storage bins to be built with vertical sides—without hoppering—which means a saving of space. The material is claimed to be discharged evenly and the level sinks regularly and without disturbance so that, with most materials, the bins never require cleaning. The discharger can also be used to give mixtures of materials in any required proportion by arranging the bins in batteries.

Clamp for pipe alignment

A pipe clamp designed to enable the speedy and accurate alignment of pipes for butt welding has recently been developed by Donald Ross & Partners Ltd. Known as the Twinner pipe clamp, it is of the split-ring type. It is placed round the pipes to be welded and positively locked by a toggle-actuated claw. The pipes are automatically aligned from the outside, while allowing the operator to adjust the distance between them to suit the weld filler.

The clamps can be used on the bench or in the field, and are made in standard pipe sizes from 2 to 12 in., with adjustment ⅛ in. larger or smaller than the specified diameter, but non-standard sizes are made to meet special requirements.

Automatic gas-pilot control device

Comprising four main components, a shut-off valve and an electromagnet (contained in the main housing) and, remote from it, a thermocouple and pilot burner unit, the new *Teddington* type TJ automatic pilot device is entirely self-contained and requires no external source of power. It can be used on either town's gas or liquefied fuel (petroleum) gases, a special burner suitable for the particular fuel to be employed being supplied.

The device is normally installed in the supply pipe to the main burner, with the thermocouple and pilot burner unit mounted in such a way that the pilot flame readily ignites the main burner. A secondary flame (heating, as opposed to ignition in the case of the main pilot flame) is also given out by the pilot, and this plays upon the tip of the thermocouple, generating sufficient potential to energise the electro-

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New Plant and Equipment

Fill in the Enquiry Coupon on page 188 for further details of the equipment and plant described in INTERNATIONAL CHEMICAL ENGINEERING.
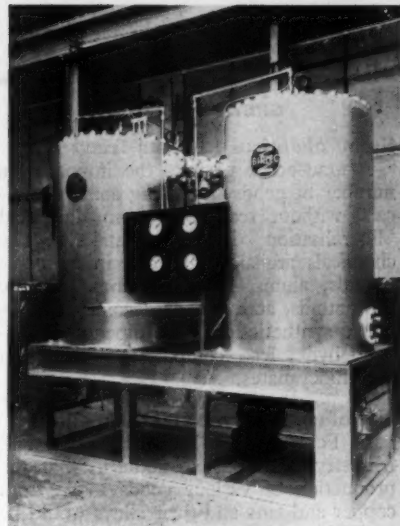
This process gas drier is designed to maintain a supply of dry air to air-operated instruments in an overseas oil refinery. Known as the Birlec-Lectrodryer, it has a capacity of 1,200 cu.ft./hr. Activated alumina is the drying agent. By using two beds of alumina, one adsorbing and the other being reactivated, continuous drying takes place while the functions of the two beds are alternated on a predetermined cycle. The alumina in this case is reactivated by steam; it does not deteriorate and has thus a practically indefinite life. The outlet gas can be dried to very low moisture content. This machine will be exhibited on the Birlec Ltd. stand at the B.I.F.

magnet in the main housing and so hold the shut-off valve in the open position.

To light the burner the push-button must first be depressed by hand. This permits gas to flow through an intermediate port to the pilot burner only and, in addition, brings an armature into contact with the electromagnet. Simultaneously, the pilot burner should be ignited. After approximately 30 sec. the thermocouple will have energised the electromagnet, which then retains the armature and so holds open the valve. The button, which up to this stage has been depressed, may now be released.

The valve remains open as long as the pilot flame is properly alight. But if the pilot flame becomes extinguished, for any reason whatsoever, then the thermocouple ceases to energise the electromagnet and the armature is released, the valve closing with a positive snap-action. Once this has happened, the valve remains closed, shutting off the gas supply to both pilot and main burners, until the device is manually restarted.

The design of this automatic pilot device provides that, in the event (say) of an interruption in its thermocouple circuit, it fails in the safe position; the gas supply to the pilot and the main burner remains closed off until such time as the fault is rectified and the operation is recommenced. The makers are the British Thermostat Co. Ltd.

World News

GREAT BRITAIN

Some chemical imports freed

A new order permits the import of a number of goods, including some chemicals, without restriction from the usual 'liberalisation' list of countries. The chemicals are alginates, aluminium compounds, alums, liquid ammonia, arsenic compounds, activated or decolorising carbons, synthetic cryolite, dipentene, formaldehyde, lead compounds, nitric acid and thiocyanates.

Use of copper, zinc and their alloys

In February the Board of Trade and Ministry of Supply announced that the production of various goods made from copper and zinc and their alloys would be prohibited. Arrangements have now been made for certain exemptions, particularly for export goods. The issue of a licence will also be considered for Government orders, for special articles where anti-corrosive material is necessary, for articles where a very small amount of metal is needed for essential parts and for work already started.

Steady rise in chemical output

According to the interim index of industrial production for the chemical and allied trades, chemical output has been increasing steadily during the past five years. Taking 1946 as 100, the figures are 105 for 1947, 120 for 1948, 123 for 1949 and about 141 for 1950.

More coal gasification tests

There is to be an expansion of the experimental work on the underground gasification of coal, according to the Minister of Fuel and Power. This follows the success of the experiments already being carried out at Newman Spinney, near Chesterfield, and other collieries (see INTERNATIONAL CHEMICAL ENGINEERING, February 1951, p. 84). For several months gas has been produced suitable for use in gas turbines generating electricity. So far, about £50,000 has been spent on the work. According to Mr. Masterman, director of underground gasification, 1 sq. mile of coal 3 ft. thick could theoretically produce enough gas to generate 25,000 kW of electricity for at least ten years.

B.O.C.'s melamine factory

A plant for producing melamine is being built by the British Oxygen Co. at Chester-le-Street; it is likely to be in operation by the end of 1951. In the meantime, the company is producing bulk samples of melamine in a pilot plant. The uses of melamine in the different fields in which it is employed are growing steadily. Already its uses for moulding powders, adhesives, laminates, paints, paper, textile treatment, etc., are considerable.

Plant exports controlled

Board of Trade licences are now necessary for the export of some types of high-pressure valves, compressors, blowers and fans, and sulphur burners and pyrites furnaces to certain destinations.

Iron and steel dearer

As a result of the recent increase in the price of coal and coke, the maximum prices for iron and steel have been increased by about 3%, according to the product. The maximum price of basic pig iron, for example, has been increased from £10 11s. 6d. to £10 19s. per ton and that for stainless steel (a) austenitic billets from from 1s. 3d. per lb. plus 10% to 1s. 3½d. per lb. plus 10%, and (b) martensitic billets from 9¼d. per lb. plus 7½% to 9½d. per lb. plus 7½%.

Electrodepositors' conference

Eleven technical papers are to be read at the Electrodepositors' Technical Society annual conference being held at Torquay from April 11-14. Three papers dealing with fundamental work are 'Studies in Discontinuities of Electrodeposited Metallic Coatings' by S. C. Shome and U. R. Evans, 'Some Applications of Interferometry to the Examination of an Electrodeposited Film' by Prof. S. Tolansky and 'Crystal Growth in Metallic Depositions' by Prof. G. I. Finch.

U.S. carton and box industry

In the recently published report on rigid boxes and cartons the Anglo-American Productivity Team advocate the installation of new and up-to-date equipment in U.K. carton factories such as is used in the U.S. This would lead to a 100% increase in output. In the U.S. machines run faster without fatigue to the operator, and productivity is in some cases 50 to 70% greater.

Copies of this report, which describes all aspects of the U.S. rigid box and carton making industry, may be obtained from the British Paper Box Federation, 127 Chancery Lane, London, W.C.2, and the Anglo-American Council on Productivity, 21 Tothill Street, London, S.W.1.

Petroleum measurement manual

An illustrated manual is shortly to be published by the Institute of Petroleum dealing with the standardisation of procedure and equipment for the sampling and measurement of bulk quantities of liquid petroleum products. It will deal with tank calibration, oil gauging, sampling, temperature measurement, specific gravity and density determinations, and calculation of oil quantities. Each part has been written so that all procedures included are applicable to either the Imperial or the metric systems of measurement.

H. W. Cremer nominated R.I.C. president

Mr. H. W. Cremer has been nominated by the Council for election as president of the Royal Institute of Chemistry for 1951-53. He is a former president of the Institution of Chemical Engineers and chairman of the D.S.I.R. committee on chemical engineering research.

Gas mains through Tyne tunnel

Inauguration of a pair of 12-in. steel gas mains through the new tunnel for cyclists and pedestrians under the River Tyne east of Newcastle marks the completion of a unique engineering project and the provision of a vital link in a ring pressure main system for the bulk handling of gas between the various works and holder stations of the Northern Gas Board in the Newcastle Division. The mains are believed to be the first ever to be used for carrying gas through a tunnel built primarily for pedestrian traffic.

Apex expands

Engineering works in Kent Road, Dartford, previously occupied by Doust & Co., marine and general engineers, have been acquired by Apex Construction Ltd., who already occupy works in Dartford.

FRANCE

Synthetic rubber project

Production of synthetic rubber from petroleum refinery gas and other raw materials normally available in France may start by 1954 if recent investigations are approved. It is estimated that up to 40,000 tons p.a. of synthetic rubber could be produced, about 25% of present natural rubber requirements. Investments required to set up such an industry are put at between 35,000 and 40,000 million francs.

Paint conference

A conference on fire-resisting and fire-retardant paints and varnishes will be held in Paris from June 3-6. It is being arranged by the Federation of European Technical Associations of the Paint, Varnish, Enamel and Printing Ink Industries, set up in January 1950 in Geneva, in collaboration with the Association Française des Techniciens des Peintures et Vernis and the Société de Chimie Industrielle. Papers will be read in the official languages of the federation, which are English, French and German.

An exhibition will also be held at the same time which will be divided into three sections covering raw materials and finished products, manufacturing plant and control apparatus, and equipment for the application of the products.

Further details can be obtained from the secretary, Fédération d'Associations de Techniciens des Industries des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale, 28 Rue Saint-Dominique, Paris (VIIIe).

AUSTRIA

New PVC plant

A factory for the production of polyvinyl chloride is at present under construction at Hallein. It is largely owned by Austrian interests, but some foreign firms, who are making available the production process, are also participating in the scheme. The factory will use only local raw materials, and will have a sufficiently large capacity to meet all local requirements. It is expected to be completed next year.

Sulphuric acid produced again

The first post-war plant to resume production of sulphuric acid in Austria goes into operation this month. Most of the country's sulphuric acid production facilities were destroyed during the war and the remainder subsequently dismantled, except for a small plant in Vienna which is under Soviet administration. Consequently, Austria has had to import 80,000 tons of sulphuric acid annually.

A new plant now being completed by the Bleiberger Bergwerks-Union in Carinthia will start production shortly at an annual rate of 15,000 tons; it is hoped to raise this figure to 30,000 tons annually by the autumn.

HOLLAND

New superphosphate factory will make sulphuric from H₂S

A 200-tons/day triple phosphate plant is being erected at Pernis near Rotterdam by the Albatross Superphosphate Co., Utrecht. A second phosphoric acid plant with a capacity of about 50 tons/day P_2O_5 is also being built. It will operate on the continuous wet process.

Although production is mainly based on pyrites, the company has already felt the effect of the world shortage of sulphur, which has caused a sharp rise in the price of their products. INTERNATIONAL CHEMICAL ENGINEERING is informed by the company that, in order to overcome possible difficulties in this direction, they are building a sulphuric acid plant operating on the wet contact system which will use hydrogen sulphide from the nearby Shell refinery. Production should amount to 15,000 tons p.a. of 78% acid. All the plants are expected to be operating by June of this year.

NORWAY

Largest aluminium factory

Plans for the erection of an aluminium factory at Sunndalsöra (see INTERNATIONAL CHEMICAL ENGINEERING, November 1950, p. 522) have been approved by the Norwegian Government. The plant will have an output of 40,000 to 50,000 tons p.a., almost equivalent to Norway's present total aluminium production capacity, and it will be her largest factory. Foreign currency for buying machinery and other equipment will be obtained through E.C.A.

and leading metal-using countries. Repayment will be made in the form of aluminium deliveries.

Liquid ammonia output up

The new liquid ammonia factory at Glomford (see INTERNATIONAL CHEMICAL ENGINEERING, April 1950, p. 186) has increased its output from 150 to 170 tons/day. The liquid ammonia is shipped to Norsk Hydro's fertiliser factory at Heröya in three 2,900-ton tankers, claimed to be the first tankers in the world specially built for this purpose. The Glomford factory cost £5,000,000 to build and employs 500 workers.

ISRAEL

New oil-cracking unit

The Haifa Oil Refineries are putting into commission a second cracking unit to provide petrol of a higher grade than that so far produced. At the same time, gases are to be produced for the nitrogen plant planned by the Israel Government.

Of the recent $35,000,000 Export and Import Bank loan for agricultural development granted to Israel several months ago, $5,000,000 is earmarked for a chemical plant to use the gases produced by the refineries.

Present output of the Haifa refineries, including petrol and other oil products, is about 20% of their total capacity, as against 15% a month ago. It is expected production will be further increased.

TUNIS

Mineral output up

Production of principal mineral ores increased during 1950, but exports were lower. Exceptions were zinc and iron pyrites, of which production declined slightly, and superphosphates exports, which expanded.

Lead mines' production for the year was 30,658 tons, or about the same as in 1938. Refiners produced 23,536 tons, compared with 19,505 tons in 1949. Lead exports comprised: France 10,059 tons, Algeria 3,318, Britain 7,112, Switzerland 1,620 and Denmark 305.

Mine production of zinc totalled 5,262 tons, compared with 6,144 tons in 1949. Exports of zinc ore comprised 5,060 tons of blende (compared with 5,534 tons in 1949) and 391 tons of carbonate (nil). Two mines reopened during the year as a result of the higher price.

Production of iron ore during 1950 amounted to 757,897 tons, compared with 711,894 tons in 1949. Exports fell from 769,046 tons in 1949 to 697,904 tons in 1950, including 464,912 tons to Britain, 99,243 tons to the U.S., 51,524 tons to Germany, 28,245 tons to Holland, 37,040 tons to Italy and 16,940 tons to France.

France remained the largest customer for phosphates in 1950, taking 452,167 tons. Britain took 230,712 tons and Italy 193,130 tons.

IRAQ

Mineral developments

It is expected that a number of British scientists and geologists will assist the Iraq Government in the exploitation of the mineral resources in the north of the country and in the search for more oil, copper, iron ore and other minerals. The Iraq Government have already allocated the necessary finance, according to a recent statement by Sayid Abdul Majid Mahmoud, Minister of Economics.

Last year deposits of copper and iron ore were found in three areas.

New sugar refineries

Three sugar refineries are to be built by a new company which has been set up by the Government to import sugar. Each factory will produce 10,000 tons of sugar p.a. It is hoped to erect the first this year.

BULGARIA

Russian equipment for fertiliser plant

The Bulgarian Government plans to erect a nitrogen fertiliser plant with an annual capacity of nearly 29,000 tons. It is scheduled to be completed by the end of 1951. Hitherto Bulgaria has depended on imports for supplies of chemical fertilisers, and this plant represents the first attempt to manufacture them domestically. It is expected that the plant will produce enough fertiliser to provide 4 lb. of pure nitrogen per acre of agricultural land. The Soviet Union is said to be supplying much of the equipment necessary for the new plant.

JAPAN

Metal and mineral imports

Planned imports of non-ferrous metals and minerals in 1951 are:

	Metric tons
Electrolytic copper	20,000
Lead	6,600
Zinc	13,100
Antimony	3,000
Nickel	1,400
Cobalt	120
Ferro-molybdenum	340
Molybdenum ore	200
Quicksilver	150
Tungsten ore	500
Chrome ore	17,000
Bauxite	163,000
Pyrites	50,000

Terrestial heat power plant

Natural steam jets will turn turbines in Japan's first power plant to use terrestrial heat which, according to the local Press, is due to start up this month. The plant, a 300,000-kW station, is at Beppu, Kyushu, Japan's southern-most island, and work on it began last July.

PAKISTAN

Lignite deposits confirmed

Recent geological surveys have indicated that the thick black mud which lies exposed for miles along the river beds of East Bengal when the waters are low is composed largely of carbon, and will burn freely if properly dried.

Last year geologists found a minimum of 9 ft. of lignite over a large area below a few feet of alluvial clay. With better equipment this year they have found 15 ft. to be the minimum thickness. This indicates that in a very accessible area there are some 200,000,000 tons of lignite.

The country's annual coal consumption is 1,920,000 tons, against its production of 400,000 tons. East Pakistan alone requires 840,000 tons p.a. and is having to import it from Poland, France, Britain and South Africa. Deliveries from India, formerly Pakistan's greatest source of coal, stopped over a year ago during the trade deadlock between the two countries. Since then East Pakistan has had a continuous coal shortage.

INDIA

Caustic soda industry expanding

The caustic soda and bleaching powder industry in India has six factories in regular production. In addition, several paper mills produce caustic soda for their own use. It is understood that four new plants are being installed or are in the planning stage.

Domestic demand for caustic soda is estimated at 55,000 to 60,000 tons annually; demand for chlorine is not expected to exceed 7,000 tons. The figures do not include production of these materials by paper mills for their own use. Capacity of the six factories is placed at 20,000 tons annually; output in the first half of 1950 was about 5,400 tons.

Before 1940, virtually all of India's requirements of caustic soda and bleaching powder were met by imports. The industry largely began and was developed during and after World War 2.

Lignite deposits

Deposits of lignite covering an area of 16 sq. miles were discovered in Madras State about 15 years ago. According to a recent report, the average thickness found in the various boreholes of the seam amounts to 22 ft. Assuming 1 sq. mile-ft. of lignite is equivalent to 1,000,000 tons, the estimate of the reserve is placed at 352,000,000 tons. The calorific value and general assay indicate that the mineral is of good quality.

Paint factory for Madras

A paint and lithopone factory with a capacity of 10 tons/day and 2,500 tons p.a. respectively is to be established at Rayalaseema, Madras. Rayalaseema has abundant raw materials used in the manufacture of paints, varnishes and lithopone. Pro-

duction in 1948 (in tons) was: barytes 17,076, steatite 244, yellow ochre 765 and red oxide of iron 1,075.

Staple fibre plant

A 15 tons/day staple fibre plant, the first of its kind in India, is expected to be in production in about eighteen months' time at Nagda, Madhya Bharat. Ancillary equipment includes a 25-ton sulphuric acid plant, a 5-ton carbon bisulphide plant, thermal power station and a cotton, spinning, weaving and finishing mill. All the plant and ancillary equipment is being supplied by Dobson & Barlow Ltd., Bolton, England. Initially imported sulphite cellulose wood pulp will be the main raw material, but it is planned to develop an indigenous source. Owners of the factory are Birla Bros.

CEYLON

Carbon dioxide project

An agreement has been concluded with the French firm Société de l'Air Liquide for the establishment of a Rs.3,000,000 manufacturing company in Ceylon. The company will erect a factory for the production of carbon dioxide for manufacturing aerated waters, dissolved acetylene, oxygen and welding equipment. The French company will provide technicians.

SOUTH AFRICA

Superphosphate shortage

The 50% cut of U.S. exports of sulphur to the Union has caused an acute shortage of superphosphates, and it is estimated that production will drop from 650,000 tons p.a. to about 388,000 tons. Farmers are now to be supplied with a new ' super raw phosphate ' fertiliser containing three parts of superphosphates to two parts of raw stock. This will facilitate the production this year of 650,000 tons of fertiliser, as the extra rock phosphate can be imported, although it will raise the price of fertiliser.

Experiments have shown that ' super raw phosphate ' is generally equal in action to superphosphate, which will only be supplied under licence in exceptional cases.

Krupp build pig-iron plant

The German firm, Friedrich Krupp, Essen, is to build a pig-iron producing plant in the Eastern Transvaal for a newly formed South African company, according to the *Rand Daily Mail*. The plant will use the Krupp-Renn process which, it is claimed, can treat low-grade and titaniferous iron ores, using low-quality coals instead of coke. Low-grade ores are not suitable for treatment in blast furnaces.

The South African company holds option contracts over a large area of iron-bearing ground near Airlie Station on the Johannesburg-Lourenco Marques railway line. Some 120,000,000 tons of iron ore exist there, it is believed.

CANADA

International Nickel chairman

Dr. John F. Thompson, president of the International Nickel Co. of Canada, Ltd., was recently elected chairman of the board of directors, succeeding the late Robert C. Stanley. He continues as president, which office he has held since February 7, 1949.

Dr. Paul D. Merica, executive vice-president and a director, was elected a member of both the executive committee and advisory committee of the company.

Good progress on ore deposits

Drilling and development work on the Labrador-Quebec iron-ore deposits has made good progress, so that well over 400,000,000 tons of high-grade open-cast ore are already in sight. Moreover, there are indications of large additional reserves, the M. A. Hanna Co. discloses in its annual report.

Shipments from the deposits will begin in 1955 and will then soon reach an annual rate of 10,000,000 tons, according to the company's president, Mr. G. M. Humphrey.

Record nickel output

Deliveries of nickel in all forms in 1950 by the International Nickel Co. of Canada Ltd. were 256,000,000 lb., a record for any peace-time year. Deliveries in 1949 were 209,292,257 lb. The company's receipts from sales of nickel, copper, platinum and other products for 1950 amounted to $U.S.228,000,000, compared with $182,806,452 for 1949. The company's current production of nickel is at maximum capacity and is expected to be larger in 1951 than in any peace-time year in its history.

U.S.A.

Improved cat crackers

The designing and building of catalytic cracking plants based on processes patented by the Socony-Vacuum Oil Co., is to be carried out by the Blaw-Knox Co. under licence. Included in these processes is *Thermofor* catalytic cracking, a modern development in oil refining designed to improve the octane number and yield (see INTERNATIONAL CHEMICAL ENGINEERING, February 1951, p. 87).

Waxes as lubricants

Production tests have shown that some waxes can be used as metal-working lubricants, according to S. C. Johnson & Son Inc., Racine, Wisconsin.

Special blends of waxes used in place of conventional lubricants allowed the drawing of stainless steel beyond its theoretical capacity, and they were also useful in aluminium-drawing. Another application was as a replacement for copper flashing on stainless-steel wire used for cold heading. Wax also extended the life of tools and dies and, in some instances, completely eliminated degreasing, it was claimed.

New catalytic cracker

A fluid catalytic cracking unit, with a reactor throughput of 60,000 barrels/day, is now under construction at Port Arthur, Texas, for the Gulf Oil Corp.

Designed and being built by the M. W. Kellogg Co., the unit is said to be not only physically the largest ever done completely by an engineer-contractor but also incorporates several recent design improvements.

According to the contractors, these accomplish a dual objective of reducing initial costs as well as improving the efficiency of the process itself. For example, from a process standpoint, the regenerator is designed to operate at higher temperatures than those normally used in existing fluid units. This will permit faster regeneration of catalyst, and also decrease the amount of catalyst that must be circulated to maintain the necessary heat in the reactor.

Although these higher temperatures require the use of a special stainless-steel standpipe from the regenerator, this is offset by the reduction in size of costly, specially formed catalyst carrier lines.

Another design change is found in the reactor, where the stripper is incorporated entirely within this vessel and above the grid rather than below. The stripper is actually a separate vessel within the reactor and, as a result of this arrangement, requires less supporting steel than designs which place the stripper outside this vessel.

A further deviation from past designs, many of which have included a flue-gas cooler and Cottrell precipitator, is the placing of the flue-gas exhaust stack on top of the regenerator. Kellogg states that this permits the elimination of a separate structure to support this outlet, and at the same time reduces the amount of piping. A special vent has been designed which permits this construction while providing for almost complete suppression of the noise which is frequently associated with the venting of flue gas from such an operation.

Shell to build resins plant

A manufacturing unit to produce the new *Epon* resins is to be constructed for Shell Chemicals Corp. at Houston, Texas. The plant consists of a three-storey process building with 4,500 sq. ft. of floor space and a full complement of auxiliary facilities. These include provisions for unloading, storage, handling and measuring of raw materials; for resin cooling, grinding, sizing, packaging and finished product storage; and a control laboratory. The process building is of semi-open construction to take full advantage of natural ventilation.

The *Epon* resins are condensation products of acetone phenol and epichlorohydrin and are used in making improved paints, varnishes and other surface coatings. They are said to have excellent chemical resistance.

Continuous aluminium casting and rolling

The continuous casting and rolling of aluminium rods is being carried out on a commercial scale by a new process which is claimed to transform molten aluminium into rods faster and cheaper than ever before, according to *Iron Age*.

The Properzi process, named after its Italian inventor, was first designed to handle lead and zinc. It employs a compact and relatively inexpensive machine which combines all the operations of a conventional rod mill. The machine, now in operation at Nichols Aluminium & Wire Co., Davenport, Iowa, costs less than $100,000 and needs a work area of only 60 × 100 ft.

Three induction furnaces melt the aluminium ingots and feed the wheel-type continuous casting machine, which produces a triangular semi-finished section. The section is automatically fed to a 15-stand continuous rod mill which rolls coiled rod. Scrap loss is under 1% and surface finish and mechanical properties are better than those achieved by conventional methods. The process is continuous and can be operated 24 hr./day. Savings in manpower are also attractive; only three men handle the entire melting, casting and rolling operation at the Nichols' plant.

Tetraethyl lead restrictions

The Petroleum Administration for Defence has ordered large manufacturers of petrol to cut their use of tetraethyl lead, an octane booster, to make more of the chemical available for aviation petrol. The order states that consumers of tetraethyl lead can purchase 1,000,000 lb. of the chemical plus 80% of what they used over this amount in 1950. Some 166 of the 220 tetraethyl lead purchasers use less than 1,000,000 lb. p.a. and so will not be affected by the order.

Safety-glass processing unit

A high-pressure autoclave process will be used to cure the glass and set the resin bond in the new safety-glass processing unit to be erected by the Guardian Glass Co., Detroit.

130-ton day soya bean plant

A new 130 tons/day soya bean processing plant is to be built at St. Joseph, Missouri, for Dannen Grain & Milling Co., by Blaw Knox Co. The extractor will be of the *Rotocel* type.

Multi-colour paint

A multi-colour paint is claimed to have been developed by the Mass and Waldstein Co., of Newark, N.J. By means of a single spray operation, products and interiors can be given a multi-colour finish, it is stated. Called *Plextone*, the finish actually appears as a network of interlaced but separate colours when applied to a surface. The company states that each colour holds its identity and gives an overall textured-colour effect like granite,

marble, etc. The only equipment needed to apply the finish is a standard type spray gun with a single pressure container. The product can be applied to wood, wallboards, plaster, metals, cement, paper and composition materials.

- MEXICO

Polystyrene production

Polystyrene in sufficient quantities for Mexico's growing plastics industry will be produced at the plant of Monsanto Mexicana S.A., first Latin-American branch of the Monsanto Chemical Co. The plant was inaugurated on February 12 at Lecheria in the State of Mexico.

PERU

Zinc refinery project

To provide power for a projected new zinc refinery, the Cerro de Pasco Copper Corp. plans to construct a new 60,000-kW hydroelectric power plant in the Paucartambo River valley, about 90 miles from Carhuamayo, involving an expenditure of at least $10,000,000. The design of the undertaking has been entrusted to a New York firm and a contract for the preliminary work awarded to the Danish firm of Christiani & Nielsen, who recently completed a similar project for the Lima Light & Power Co. The undertaking is expected to be completed in four years.

VENEZUELA

More cement capacity

Production of cement in Venezuela in 1953 will be sufficient to meet domestic consumption if expansion plans of the industry are realised. Output in 1950 amounted to 500,000 tons, as against 300,000 tons in 1949.

One of the important cement manufacturing concerns, Compania Venezolana de Cemento, maintains three plants which at present are operating at capacity. The company is investing 20,000,000 bolivares in plant expansion and new equipment ordered for the company's Pertigalete plant has already been installed, while machinery for the other two plants will arrive in the country shortly.

Company officials predict that the three plants will be producing cement at the rate of 12,000,000 bags by the end of 1953 from the present level of 2,000,000 bags (93½ lb. each).

YUGOSLAVIA

Coal-tar chemicals industry

Yugoslavia is now producing various coal-tar products which she formerly obtained from abroad, including *ortho*, *meta* and *para* cresol and various phenolic and acidic resins, at the Katran factory in Croatia. The extraction and distilling plant was partly manufactured in Yugoslavia and partly obtained as reparations from Germany. The extraction plant started operations at the end of 1949, while

the distillation section has been working since the end of last year.

The plant will also be able to process coal-tar products from Yugoslavia's first coking ovens at Lukavac, which are due to start operation at the end of this year. The new plant will also be capable of refining and separating various mixtures of organic oils.

The resulting products are being used in Yugoslav electric light bulb, dyestuffs and plastic factories, and are already estimated to have saved the equivalent of 7,000,000 dinars in foreign exchange. Savings this year are estimated at 14,000,000 dinars. The process involves steam distillation and was evolved by Yugoslav technicians.

New chemical plants

The chemical industry in Yugoslavia has built 36 plants in the past four years, according to the local Press.

Synthetic detergents produced

The Ljubljana Institute for Industrial Research is producing petroleum-based detergents. With the exception of benzene, all necessary raw materials are available from home sources. Eventually it will not be necessary to import benzene and production will then be entirely from domestic materials.

Meetings

Institution of Chemical Engineers

April 11-15. Graduates and Students' Section, 1951 Convention. 'Chemical Engineering—Opportunities in Specific Industries,' Nutford House, Brown Street, London, W.1.

April 28. North-Western Branch. 'Chemical Engineering as Applied to the Building of a Petroleum Chemicals Plant,' by H. E. Charlton, 3 p.m., Reynolds Hall, College of Technology, Manchester.

Society of Chemical Industry

April 10. Chemical Engineering Group. 'Man and the Chemical Industry: A Consideration of Physiological Adapta-

bility,' by Dr. M. W. Goldblatt, 5.30 p.m., Burlington House, London, W.1.

April 19. Road and Building Materials Group. 'Problems Involved in Obtaining Colour and Texture in Concrete Surfaces,' by J. G. Wilson, 6 p.m., Lecture Hall, Institution of Structural Engineers, 11 Upper Belgrave Street, London, S.W.1. Joint meeting with the Institution of Municipal and County Engineers.

May 7. London Section. 'The Growing of Synthetic Crystals,' by L. A. Dauncey and J. E. Still, 6.30 p.m., London School of Hygiene and Tropical Medicine, Keppel Street, W.C.1.

May 8. Chemical Engineering Group. 'Economic Reactor Design,' by G. T. Meiklejohn and R. C. Snell, 5.30 p.m., Burlington House, London.

Chemical Society

April 26. 'Studies in Infra-red Spectroscopy,' by Dr. W. T. H. Williamson, 7.30 p.m., Marischal College, Aberdeen. Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry.

British Association of Chemists

April 18. London Section. Symposium, 'Technical Service: A New Branch of Applied Chemistry,' by C. W. Tod, R. C. Tarring and R. J. Smith, 7.15 p.m., Kent Room, Caxton Hall, London, S.W.1.

Institution of Mechanical Engineers

April 13. 'Steam Piping for High Pressures and High Temperatures,' by R. W. Bailey, 5.30 p.m., Storey's Gate, St. James's Park, London, S.W.1.

Institute of Petroleum

April 11. I.P. Standardisation. Papers by members of the I.P. Standardisation Committee, 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

April 17. 'Alloyed Oils,' by R. D. Streeton and A. C. Mauchan, 6.30 p.m., the Engineers' Club, Albert Square, Manchester.

May 9. Symposium, 'Combustion Reactions in Relation to Gas Turbine Prac-

tice,' 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

Institute of Fuel

April 9. 'Atomic Energy,' by J. Diamond, 6.30 p.m., King's College, Newcastle.

April 25. Final conference in 'A Study of Drying,' 10 a.m., Institution of Electrical Engineers, Victoria Embankment, London, W.C.2.

Fertiliser Society

April 12. 'Research on Phosphate Fertilisers at the Chemical Research Laboratories of the D.S.I.R.,' by F. R. Reynolds, 11 a.m., Chemical Research Laboratories, Teddington.

May 10. 'Cliff Quay Works,' by R. F. Parker, 11.45 a.m., Ipswich. Visit to Cliff Quay works, Fisons Ltd. Also Annual General Meeting.

Royal Society of Arts

April 25. 'Safety in Mines,' by Sir Andrew Bryan, 2.30 p.m., Royal Society, Burlington House, Piccadilly, W.1.

Electrodepositors' Technical Society

April 18-21. Annual conference, Grand Hotel, Torquay.

May 1. 'The Protective Quality of Nickel-Chrome Deposits,' by S. Wernick, James Watt Memorial Institute, Great Charles Street, Birmingham 3.

Institute of Refrigeration

April 19. 'Some Considerations on the Heat Pump,' by P. B. H. Brown, 5.30 p.m., Institution of Mechanical Engineers, Storey's Gate, St. James's Park, London, S.W.1.

Institution of Works Managers

April 10. 'The Value of Statistical Methods of Control,' by P. Lyle, 7 p.m., Grand Hotel, Birmingham.

Society of Instrument Technology

April 10. 'pH Measurement,' by A. D. R. Lauchlan, 7.30 p.m., College of Technology, Manchester.

Institute of Welding

April 11. Annual General Meeting and film show, 7 p.m., Reynolds Hall, College of Technology, Manchester.

Society of Glass Technology

April 12. 'Glass-Working Machinery,' by British-Hartford Fairmont Ltd., 6 p.m., Gas Showrooms, Radiant House, St. Helens, Lancashire.

Manchester Statistical Society

April 20. Industrial Group. 'Production Statistics,' by R. G. Hooker, 6 p.m., Albert Hall, Peter Street, Manchester.

Oil and Colour Chemists' Association

April 23. Annual General Meeting, preceded by film, 'The Film of Paint,' 7 p.m., Royal Station Hotel, Kingston-upon-Hull.

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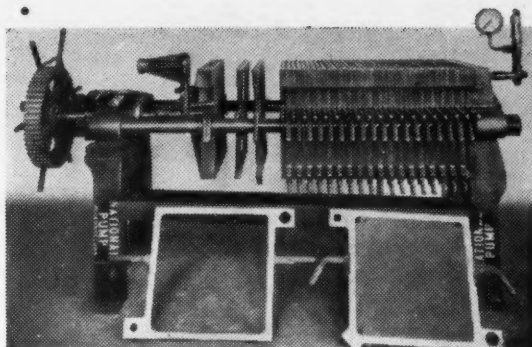
Illustration shows side view of "National Pump" 24" × 24" × 24 Chambers Plate-and-frame Model Filter Press, as used for the Food Processing, Chemical, Mining, and Oil Refining and Allied Industries.

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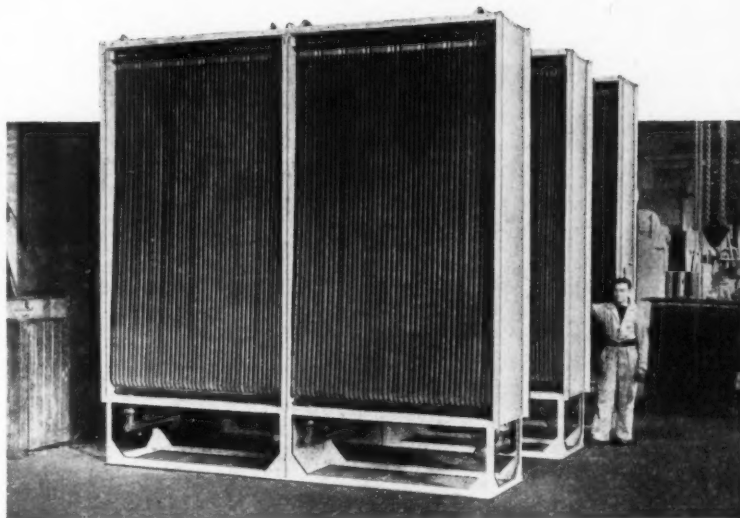
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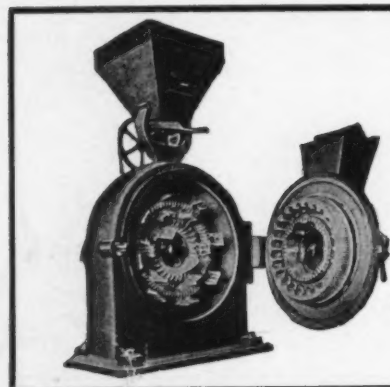
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